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STRENGTH AND CREEP CHARACTERISTICS OF CERAMIC

BODIES AT ELEVATED TEMPERATURES

By M. D. Burdick, R. E. Moreland, and R. F. Geller

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SUMMARY

A total of 109 tests has been made to evaluate certain characteristics of six ceramic oxide bodies for high-temperature applications, especially as turbine blades. Primary emphasis was placed on determinations of strength and resistance to creep under tensile stress at elevated temperatures. Additional data for these bodies on bulk density, linear thermal expansion, modulus of elasticity, and effect of water vapor on strength are given.

Bodies 4811C and 16021T, those of highest beryllia content, showed no loss of strength after 10 cycles of quenching from 1700° F to an air blast at room temperature. The high-zirconia bodies showed either loss of strength after this treatment or cracked before its completion.

Bodies 4811C and 16021T had bulk densities of 3.0 grams per cubic centimeter. The densities of the other four bodies ranged from 3.8 to 4.9 grams per cubic centimeter.

Approximate values for Young's modulus of elasticity, calculated from the tension test data, ranged from 36×10^6 psi to 15×10^6 psi at temperatures between 1500° and 1900° F. Values for Young's modulus, calculated from the bending test data for temperatures between 1500° and 1900° F, ranged from 36×10^6 to 8.8×10^6 psi.

Creep rates as low as 3.0×10^{-4} percent per hour at 17,000 psi and 1800° F, 16.2×10^{-4} percent per hour at 15,000 psi and 1900° F, and 12.7×10^{-4} percent per hour at 6,000 psi and 2100° F were observed. Strengths of 18,000 psi at 1800° F and 15,000 at 1900° F seem assured for some bodies. The maximum stresses sustained for at least 160 hours by the four strongest bodies ranged from 17,000 to 18,000 psi at 1800° F and from 4,000 to 16,000 psi at 1900° F. Body 4811C was equal or superior to the other bodies in any property determined.

The maximum stresses sustained for an appreciable length of time at various temperatures by the four strongest bodies are tabulated as follows:

Temperature (°F)	Maximum stress (psi) for body -			
	358	353	151	4811C
1500	13,000	13,000	12,000	14,000
1700	14,000	13,000	13,000	14,000
1800	17,000	18,000	18,000	18,000
1900	8,000	4,000	15,000	16,000
2100	------	------	------	6,000

INTRODUCTION

The revolutionary developments during the past 30 years, and especially the last 10 years, in both design and efficiency of power plants have emphasized the need for materials of great strength and durability at very high temperatures. This is a natural consequence of the fact that, in any device for the conversion of heat energy into work, the efficiency of that device may be increased by increasing the temperature differential between the beginning and end of the conversion.

Design engineers soon projected their plans into regions of temperature and stress far beyond the potentialities of known metallic alloys. This led to a survey of metal compounds, especially the oxides, silicates, carbides, and related combinations peculiar to ceramics. Such possible applications for ceramics in power plants as the jet engine were discussed by Conway (see reference 1), but there was very little data in the literature upon which to base specific designs. Some work had been done on feldspathic bodies (see references 2 to 8) which contain a bond of glass and, consequently, have limited use at elevated temperatures (see reference 9). Several reports were available also in the German literature (see references 10 to 12) on small specimens of simple oxide bodies with extraordinary resistance to mechanical stress at temperatures above 1800° F. These bodies had been matured at such high temperatures (about 3450° to 3550° F) that their successful production commercially in the United States seems to be only a remote possibility.

Consequently, when the National Bureau of Standards was requested, by the National Advisory Committee for Aeronautics in 1944, to obtain engineering data on ceramic bodies in tension at elevated temperatures, it was thought advisable to begin the work on bodies which could be produced with available industrial facilities. Leading ceramic concerns were requested to submit specimens for preliminary tests of bending at elevated temperatures and of resistance to thermal shock. Also, six promising bodies were selected from a number developed at the National Bureau of Standards during the course of an investigation which was later reported in part in 1946. (See reference 9.) The results of the strength in bending at 1800° F and of relative resistance to thermal shock were incorporated in another paper published in 1946. (See reference 13.) Based on these preliminary tests, a comprehensive investigation was begun on the strength and creep in pure tension of the six previously selected NBS bodies, supplemented by a few comparison trials on two commercial bodies. The results of the tests in tension are given in reference 13. The present paper summarizes the previously published data and gives the later results. It was not feasible to reproduce all the large quantity of data recorded during this investigation; therefore, not all the material discussed in the text is presented in the tables and figures of this paper.

GENERAL CONSIDERATIONS

The advantages and limitations of ceramics for service at high temperatures are discussed in references 9 and 13. Briefly, the advantages are their high resistance to deformation, fusion, and chemical changes and their relatively low specific gravity. The disadvantages of ceramics, when compared to metals and metallic alloys, are their relatively low thermal conductivity and their brittleness. Consequently, their resistance to mechanical impact and to rapid fluctuations in temperature leaves much to be desired. As a generalization, research in ceramics should be aimed toward the development of materials with high thermal conductivity, low thermal dilation, high ratio of strength to modulus of elasticity, and high mechanical strength at elevated temperatures which, in this case, means temperatures above 1800° F.

High mechanical strength involves high resistance to softening. Full advantage of this property is obtained by so formulating the bodies as to insure the practical elimination of a glassy phase and the development

either of a single crystal from which the entire article can be formed
or of a very fine crystal structure in which the individual crystals
preferably are not larger than about 5 microns. The modulus of
elasticity, a value expressing the resistance to elastic deformation,
usually increases with increase in strength. It is also unfortunate that
the refractory oxide materials commercially available in nearly pure form,
such as crystalline thoria (ThO_2), magnesia (MgO), zirconia (ZrO_2),

calcia (CaO), beryllia (BeO), and alumina (Al_2O_3), have high thermal expan-
sions, the coefficients ranging from about 9×10^{-6} for alumina to 15×10^{-6}
for magnesia in the interval from room temperature to $3092°$ F $(1700°$ C$)$.
(See reference 14.) Of the better-known low-expansion materials, such as
silica glass, silicon carbide, beryl, cordierite, zircon, and zirconium
phosphate, none is outstanding in both strength and refractoriness. They
no doubt will, however, be indispensable for some applications. Zircon,
for example, has better than average strength and resistance to thermal
shock, but would not be suitable for service at very high temperatures
because it breaks down at about $3225°$ F to form zirconia and a liquid high
in silica. (See reference 14.) In fact, some investigators claim the
breakdown may occur as low as $2730°$ F in the presence of certain
impurities. (See reference 15.) There is no known ceramic material,
stable in oxidizing atmospheres at very high temperatures, that can even
approximate beryllium oxide in high thermal conductivity. For an arbi-
trary scale on which the conductivity of beryllia is 10, relative values
would be about 3 for a typical high-alumina spark-plug insulator, 2 for
magnesia and thoria, and 1.5 for zirconia. (For method of determination,
see reference 9.) The relative value for body 4811C, described in this
report, would be about 9. Assuming that 3 will apply to the alumina-type
insulators for which Riddle has published absolute conductivity values
(see reference 16), the thermal conductivity of body 4811C can be cal-
culated, in British thermal units per square foot per hour per degree
Fahrenheit per inch thickness, to be approximately 60 at $100°$, 72 at $400°$,
and 85 at $800°$ to $1600°$ F.

WATER-VAPOR TEST

There is sufficient information in the literature (see references 18
to 21) regarding the possible deleterious effects of water vapor on
ceramic bodies at elevated temperatures to justify its consideration in
connection with porcelains subjected to products of combustion containing
such vapor. Crystalline magnesia, for example, is attacked by water vapor
at room temperatures. Therefore, the solution or disintegration effects
at elevated temperature may be appreciable in bodies containing uncombined
magnesia. A few tests of an empirical nature were made, using the
following method:

A fused-silica tube was heated by means of the conventional furnace used for organic-combustion tests. One end of the tube was connected with a flask in which water was boiled vigorously during the experiment. After the tube was heated to 1100° F, a specimen in the form of a bar was introduced slowly into the heated zone. The temperature was then raised to 1700° F and maintained for 5 hours during which time the water vapor passed over the specimen. At the end of this time the boiling of the water was discontinued and the furnace allowed to cool to 1100° F overnight. The following morning the specimen was removed slowly from the furnace. The introduction and removal of the specimen occupied about 5 minutes and it is believed that harmful effects from thermal shock were avoided. The weight of the bar and the strength in bending were then determined and compared with corresponding values for untreated bars.

The results of the vapor test are given in table 1. They indicate that both the commercial compositions showed considerable decrease in strength. Neither of the high-beryllia bodies (4811C and 16021T) was affected. It is interesting that specimens of the high-zirconia body (353) containing 19.6 weight percent of magnesia were stronger than the comparable untreated bars and that the specimens (358) containing 9.8 weight percent of magnesia were weaker. The results indicate that some type of simulated-service test should be used when "screening" experimental compositions designed for application at high temperature involving exposure to water vapor.

TENSION TEST

Specimens and Apparatus

The compositions developed at the National Bureau of Standards that were selected for tension tests are described in table 2. The shape of the specimens, round in cross section and approximately 0.3 inch in diameter throughout the gage length, is shown in figures 1 and 2. The adapters (see fig. 2) for applying the stress were $8\frac{1}{2}$ inches long,

$1\frac{3}{16}$ inches in diameter, and were made of body 358. Ceramic adapters were used because calculations based on the data available indicated that high-temperature alloys would not have sufficient strength at the maximum temperatures and stresses likely to be obtained in some of the tests. Adapters and test specimens were fabricated under contract by a cooperating industrial concern, using hydrostatic pressure applied by means of rubber molds.

The thread fit between specimen and adapters was made loose intentionally to permit a liberal filler of cement. This cement was made of very fine crystalline-alumina and sodium-silicate solution. When the

threads were coated with this cement paste, the specimen was screwed into
the adapters and the assembly alined in a jig until the cement had set.
Infrared lamps were used to expedite the setting. To assure complete
dehydration of the cement, the assembly was next heated in a furnace
at 1800° F for 2 hours under a total superimposed load of 20 pounds. If
examination then showed the assembly to be in good alinement and the
adapters to be free from cracks, the gages were cemented on, as shown in
figure 1. These gages, similar in design to those used at the National
Bureau of Standards for measuring creep of metals, are a modified form
of the gage described by Fellows and co-workers. (See reference 22.)
They were fabricated from 90-percent-platinum and 10-percent-rhodium
tubing and wire.

Two thermocouples (platinum to platinum and 10 percent rhodium) were
mounted, one on each adapter, extending from the outside of the furnace
to one end of the gage length. A groove was provided in the adapters
(see fig. 2) for this purpose. The entire assembly as mounted in the
furnace and the method of loading are indicated in figure 3. As this
figure indicates, there were two concentric heating coils. The outer coil
was in one unit, connected directly across a regulated 112-volt power
source, and consisted of 16-gage, 80-percent-nickel and 20-percent-
chromium resistance wire. The inner coil was in three units, each of
which was connected to the output of an adjustable-ratio autotransformer;
in addition, the center unit was subject to automatic control. In seven
furnaces, this inner coil was made of 20-gage, 80-percent-nickel
and 20-percent-chromium wire; in five furnaces it was made of 22-gage,
80-percent-platinum and 20-percent-rhodium wire. By proper manual
setting of the three transformers, the automatic controller maintained
a predetermined average air temperature over the entire gage length to
within ±5° F. Besides the two thermocouples for reading the temperature
at each end of the gage length, two others of platinum to platinum
and 10 percent rhodium were inserted horizontally in the plane of the
windows so that the ends nearly touched the specimens at mid-gage
length. One of these was used for the automatic control of the center
heating unit, the other was connected to a multiple-point temperature
recorder. In order to have a record of the time of any specimen failure
occurring outside the regular working hours, a switch located under the
loading beam at the adjustable fulcrum support (fig. 3) was closed when
the beam fell. The closing of this switch introduced a shunt across the
recorder-thermocouple circuit and caused the recorded temperature to be
about 200° F below the true temperature.

A Gaertner extensometer-viewing device for observing the length
changes has been described. (See reference 23.) Measurements can be
made with a precision of 1 micron.

Procedure·

Four general methods of test were followed, the first two of which
may be described as "step testing." They are:

Method 1.- The specimen was heated gradually to the test
temperature and a stress considered to be well below the tensile
strength of the specimen was applied. After observing length
changes for some predetermined time, the stress was increased.
Length changes were observed again, whereupon another increment
of stress was applied and so on to rupture. It was customary to
increase the stress in 1000-psi increments at about 200-hour
intervals.

Method 2.- The original temperature and load conditions were
essentially as described for method 1. In this method, however,
the load remained constant and the temperature was raised, usually
in 100° F steps at about 200-hour intervals. In some cases, the
specimen had not ruptured after prolonged holding at 1800° F. When
this occurred in a furnace in which the temperature could not be
raised safely above 1800° F, the test was continued by increasing
the load in steps while maintaining the 1800° F temperature.

Method 3.- After holding the specimen at 1800° F under a stress
of 4000 psi for 48 hours, stress was increased at the rate of
about 1200 psi per minute to failure. The load was increased by
flowing shot ·into a bucket.

Method 4.- The specimen was held at· constant stress and
constant temperature to failure, the purpose being to obtain
curves of stress against rupture time which could be compared with
similar curves for metal alloys. However if, after 1000 hours, the
creep rate was less than 1.0×10^{-4} percent per hour and the total
extension was less than about 0.40 percent, the stress was increased.
The increment of stress depended on the creep rate obtained.

Several developments interfered with the successful completion of
some of the tests: (a) Failure sometimes occurred in the adapter or
in the threaded head of the specimen; (b) on a few occasions the
electrical power supply was interrupted; (c) after prolonged holding
(usually over 2000 hr), the base-metal thermocouples used in the first
few tests became inoperative; or (d) the reference points became obscured
as a result of grain growth in the platinum.

RESULTS

Step Tests

This section is devoted to those results obtained by test methods 1 and 2. Typical data, presented in figures 4 to 21, are grouped and discussed according to body compositions. For some tests the curves show irregularities, and even an apparent contraction may be indicated under the lowest stresses. Deviations of not over 0.004 percent from a mean curve are, however, considered to be within the over-all reproducibility of a length-change determination. Total extensions include the elastic deformations which occur upon application of a load increment and also the slowly recoverable "viscous-elastic effects" described in reference 8. In several experiments the elastic and the viscous-elastic recoveries were observed upon removal of the load.

Those tests that were concluded successfully, in the sense that failure came in the constricted portion of the tension specimen, are summarized in table 3. This does not mean that the tests culminating in failure of the apparatus, or failure at some other location in the assembly, were without informative results. Test F8-4, for example, (see fig. 4) gives the elongation behavior for body 358 at 1800° F under stresses ranging from 4000 psi to 15,000 psi during a total test period of 2247.5 hours, followed by observation of strain recovery during 193 hours.

In the discussion of creep, a rate of 0.0001 percent per hour, or less, will be referred to as very low and a rate of over 0.0010 percent per hour will be considered as very high.

Body 358.- Of the 17 step tests made on the composition, body 358, 14 were continued sufficient lengths of time to give creep results and, of these, 8 were completed successfully. (See table 3.)

Results at room temperature, at 1500° F, at 1600° F, and at 1700° F show no significant elongation or positive creep following the elastic deformation. Theoretically, the average creep rate should increase regularly with increase in stress increments and, actually, it probably does. The rates are so low, however, that experimental error can easily account for irregularities such as the comparatively high maximum observed creep rate at 14,000 psi in test F8-3. (See table 3.) A "negative creep," or contraction, was observed in some cases when the specimens were under the initial stresses. This contraction was noted in many of the tests and frequently exceeded the probable experimental error. (The over-all experimental error of a series of length-change

readings will be discussed in connection with body 353.) The conclusion
is that this contraction is real, but the available data do not suggest
an explanation for it. It may be noted, also, that for body 358 the
stresses at failure given in table 3 for temperatures below 1700° F are
lower than the values obtained at 1700° and 1800° F. It is believed
that the specimens are not·actually weaker at the relatively low tem-
peratures. The explanation is offered that, ·at higher temperatures,
the specimens and adapters may adjust themselves to certain unmeasured
variables, such as .slight deviations from straightness or misalinements
in the assembly, thereby effecting a better stress distribution.

Creep rates continue low at 1800° F in test F8-4 (fig. 4) and it
was only at 15,000 psi that the rate exceeded 0.0005 percent per hour.
The total elastic strain prior to the load removal was. 0.088 percent;
the recovery was 0.134 percent and was practically complete after
193 hours. Obviously there is some error in the determinations to
account for a recovery nearly twice the observed elastic strain. This
error probably lies in the assumption that the elastic strain was com-
plete within 60 minutes after a stress increment rather than in the
measurements themselves. Sixty minutes may not be sufficient to cover
the viscous-elastic strain (see reference 7) which was recovered during
the 193 hours. It may be seen from the curve that 0.088-percent recovery
took place in only about 15 hours.

In test F11-5 (fig. 5) the recovery (0.057 percent) again was about
twice the measured elastic strain (0.025 percent), but 0.025-percent
length change was recovered within 1 hour; whereas nearly 200 hours was
required for total recovery. This test was completed successfully. The
pronounced rise in creep rates at 1900° F and various stresses, compared
with the rates at 1800° F, is shown by the curves in figure 6.

Test F11-6 (table 3) was conducted in two stages. In the first,
according to method 2, the maximum temperature was 1950° F. In the
second, according to method 1, the stress at failure, 18,000 psi, is
the highest obtained for body 358 and one of the highest obtained in this
investigation at 1800° F. Apparently the pretreatment at 1950° F had not
harmed the specimen.

The results for test F12-7 are shown in figure 7. This test also
was conducted in two stages, with a constant stress used throughout. In
the first stage the temperature was increased in increments to 1950° F,
curves A in the figure. The temperature was then dropped to 1700° F and
again raised in increments to 1950° F. The lower creep rates noted in
the second series are shown by the curves B. To facilitate comparison with
the curves A, obtained in the first series, the ordinates were adjusted
so that the starting point of the two curves coincided. This lowering of
creep rate was noted in many other tests and will be discussed later.

Body 353.- Seven step tests (table 3) were completed successfully out of the fourteen undertaken with specimens of body 353.

The creep rates at 1500°, at 1700°, and, in test F8-2, at 1800° F are in general all very low. (See table 3.) However, the values obtained at 1800° F in test F1-11 (fig. 8) are believed to be the better data for this temperature. The stress at rupture of 18,000 psi is as high as the highest obtained in this study at 1800° F. Also, the tendency for the creep rate to increase after prolonged holding, as shown by the curves for stresses of 16,000 and 17,000 psi (fig. 8), is unique. Usually, the creep rate continued to decrease even after 1000 or more hours of holding. This will be shown later in the curves of stress against rupture time. The strain recovery shown in figure 8 equaled 0.086 percent and it was regained in 92 hours, but 0.034 percent (equal to the measured elastic strain) was recovered in about 7 hours.

In tests F12-4, F10-5, and F9-11, for which detailed data are not presented, an attempt was made (unsuccessfully in the first two) to check by direct measurement the length changes obtained with the extensometer-viewing device. Two pairs of platinum discs, bearing reference marks ruled in the Length Section of the National Bureau of Standards, were cemented to the specimen in the same plane as the two platinum gages. Direct measurements of the length between reference marks were then obtained before and after the treatment in the creep furnace. The results show satisfactory agreement:

Gage	Length change by -		Difference in measurements (μ)
	Extensometer measurement (μ)	Direct measurement (μ)	
Right	116	111	5
Left	109	112	3

In general, the data for body 353 are straightforward and subject to the same comments made for body 358 at comparable temperatures.

Body 163.- Only seven step tests were made on the composition, body 163, and four were completed successfully (table 3). At 1500° and 1700° F the creep rate was very low for all stresses up to 16,000 psi.

Even at 20,000 psi (fig. 9), it averaged less than 0.0002 percent per hour for the first 71.5 hours. This 20,000-psi stress at rupture was also the highest observed at 1700° F in any of the tests.

The data from tests at 1700° and 1800° F (table 3, tests F10-4 and F7-6) show the increase in creep with temperature. At the latter temperature a stress of 7000 psi produced more creep per hour than did 20,000 psi at 1700° F. Further comparison of the data from test F10-4 (fig. 9) with the data in figure 10 (test F10-6) shows the effect of increasing the temperature to 1900° F. At this temperature a stress of 3220 psi produced a higher rate of creep than did 11,000 psi at 1800° F. The data in figure 10 and table 3 show the very high rates of creep to be expected at 1900° to 2000° F. It is interesting that the specimen in test F12-5 withstood 2000° F and 4000-psi stress for 309 hours before failure, even though the creep rate was 0.007 percent per hour. (See fig. 11.) The data at 2000° F and 4000 psi are interesting also because this is one of the rare instances throughout the entire investigation in which the creep rate increased, rather than decreased, after having been held for 100 or more hours. The creep rates of this body at various temperatures, as compared with those of two other bodies for which comparable values are available, are indicated by the curves in figure 11.

Body 151.- Fourteen step tests were started with specimens of body 151. Of these, 10 were of sufficient duration to provide creep data (typified by figs. 12 to 15) and 8 were completed successfully (table 3).

The data for temperatures up to, and including, 1700° F show negligible, or very low, creep. In view of the 19,000-psi stress at rupture at 1600° F (table 3) and the 18,000-psi stress at rupture at 1800° F (fig. 12), it seems a reasonable assumption that some undetermined, but detrimental, condition in the test assembly caused the relatively low resistance to rupture at room temperature and at 1700° F.

The families of curves in figures 12 and 13 are among the most regular obtained in this investigation and so afford a good comparison of relative creep at 1800° and 1900° F (fig. 6); the stresses at rupture are among the highest observed at these temperatures. As mentioned previously in the discussion of bodies 358 and 163, the creep for all bodies is typically very high at temperatures of 1900° F and above when under stresses approaching those causing failure. It is interesting, however, that the creep rates for body 151 (the highest in beryllia content of the so-called zirconia bodies 358, 353, 163, and 151) are next higher than those for body 4811C.

The tests for which results are given in figures 14 and 15 were obtained by maintaining a constant and relatively low stress and increasing the temperature in steps until failure occurred or an excessive creep rate was obtained. Figures 14 and 15 contain the data for the curves in figure 11 which illustrate the comparative effect of 4000- and 6000-psi stresses in the temperature range 1800° to 2050° F. Although body 151 compares favorably with 4811C in strength, the curves in figure 11 show the creep resistance of body 151 to be considerably lower than that of body 4811C.

Body 16021T.- Twelve step tests were started on specimens of body 16021T. Of these, 10 produced creep data and 9 were completed successfully (table 3).

In common with the other compositions tested, body 16021T shows no definitely measurable creep at room temperature, 1500°, or 1600° F, and the elastic strain constituted practically the entire length change. In fact, the creep rates were low in all the tests, including those at 1800° F (fig. 16). However, this composition had the lowest strength of the six investigated. Each of the four tests in which failure occurred at 1800° F was completed successfully, but the strengths were only 6000 and 7000 psi (table 3). This is very interesting in view of the fact that body 4811C, which most nearly resembles 16021T in composition, was on the average the strongest and also the most resistant to creep.

Body 4811C.- Eleven of the fifteen step tests undertaken with body 4811C were satisfactory for creep data (typical curves in figs. 17 to 20) and eight were completed successfully (table 3).

The highest average creep rate for an extended period of time at 1500°, 1600°, and 1700° F was only 0.0001 percent per hour for the test conditions of 18,000 psi and 1600° F. The 20,000-psi stress at rupture (fig. 17) was the highest obtained for any body at 1600° F. The recovery shown in figure 17 was 0.018 percent; the observed elastic strain was 0.013 percent. In test F9-9 the corresponding values were 0.016 and 0.023 percent, respectively.

The test recorded in figure 18 was completed successfully and the 18,000-psi stress at rupture was as high as any obtained at 1800° F. The data in figure 18 (test F8-5) were used in figure 6 to compare creep rates at 1800° F with those at 1900° F. Values for 1900° F were taken from the two tests presented in figure 19. The strength at rupture of 16,000 psi (test F11-7, fig. 19) was the highest obtained at 1900° F.

The data from tests F10-7 and F11-2 (fig. 20 and table 3) were
used in figure 11 to show the relative creep at various temperatures
under stresses of 4000 and 6000 psi. As figures 6 and 11 indicate,
body 4811C had the highest resistance to creep of any tested and it had
the highest strength at all temperatures except 1700° F. It is probable
that the 14,000-psi strength obtained at 1700° F is not representative of
the true potentialities of body 4811C. Also, the 6000 psi at 2100° F and
4000 psi at 2230° F were obtained in the only tests completed successfully
at these temperatures (table 3).

Short-Time Tensile Tests

All the short-time tensile tests were made with the specimens at
1800° F. Eight tests were conducted without prestressing. The furnace
was brought to equilibrium at 1800° F with the test assembly in place
but with no imposed load. Stress was then applied at the rate of about
1200 psi per minute. The results have been reported in reference 13 and
are repeated in group A of table 4. Eleven additional tests were made
in which prestressed specimens were used in accordance with the described
method 3. Because of the shortage of specimens, it was necessary to use
several that had been heated and stressed previously in other tests.
References to these treatments and the data from the short-time tests
are given in group B of table 4.

The most prominent comparison observed in table 4 is the location of
fracture in the unprestressed test assemblies (group A) and the
prestressed (group B). In the former group, 7 out of 8 failed outside
the constricted portion of the specimen; in the latter, only 1 out of
11 fractures was unsatisfactory. This appears to support the idea,
mentioned earlier in connection with body 358, that testing under stress
at elevated temperatures may effect better stress distribution in a test
assembly. However, the values in group A show that body 163 has a
potential strength of at least 10,000 psi in a short-time test at 1800° F,
and body 4811C is indicated to have a corresponding strength of at least
19,000 psi. This potential strength of body 4811C is supported by the
values in group B.

According to the limited data in table 4, the arrangement of the
four strongest bodies in the order of increasing strength at 1800° F
would be: 16021T, 358, 151, and 4811C.

Stress-Rupture Test Results

A total of 23 tests was made according to method 4 in an attempt to
obtain values upon which the curves of stress against rupture time could
be based. Such curves show the relation at a given temperature between

a series of constant, initially applied, stresses and the time required
for each stress to cause failure. They are used for evaluating metals
and metallic alloys. (See fig. 4 in reference 24.)

When the laboratory work of the investigation was closed, three tests
were still in progress. Only eight tests had been completed successfully;
that is, the specimens had fractured in the gage length. However, one
curve was obtained (fig. 21). It is based on the very limited data for
body 16021T given in table 5. In none of these stress-rupture tests was
there observed the so-called third stage of creep, in which the rate
continually increases and which inevitably must lead to failure. Rather,
the values in table 5 show that the rate of elongation under a constant
stress continued to decrease even after 1000 hours or more.

The statement was made on page 15 of reference 13 that a curve of
stress against rupture time for a ceramic body may not be obtainable
because the strength and creep behavior may be altered by stress-
temperature tests. According to this hypothesis, a specimen that has
been under stress at, for example, 1800° F for 1000 hours may not be the
same structurally as it was at the beginning of the test. The longer the
test continues the greater the change, as evidenced by the continued
decrease in creep rates with time of stress (table 5). The effect of
temperature and stressing on creep is shown in table 6.

Another indication of structural change is provided by typical thin
sections. In the high-beryllia bodies (16021T and 4811C), the changes
were not sufficiently pronounced to be visible, but changes were observed
in sections of the bodies high in zirconia content. With white trans-
mitted light, the cubic zirconia crystals in thin sections of the bodies
that had undergone considerable creep appear light tan in color and do
not extinguish completely. It was assumed at first that this was a
strained condition brought about by a combination of stress and tempera-
ture. However, specimens of bodies 151 and 353 showed the same appear-
ance after having been held for 14 days at 1900° F with no superimposed
load. X-ray patterns showed partial reversion of the cubic zirconia to
the monoclinic form. In these thin sections, the zirconia crystals
appear also to have undergone some marginal alterations and, as a result,
to have lost the sharp crystal fractures characteristic of the specimens
not subjected to a prolonged creep test.

Modulus of Elasticity

Values of the modulus of elasticity were computed from the data
collected during the step tests. The increments of stress were usually
1000 psi and the resulting increments of strain were between 2
and 9 microns over the 100-millimeter gage length. These values for the

modulus of elasticity could not be very precise when the reproducibility·
of a single length measurement was of the same order of magnitude as the
strain that was measured. In several cases, additional tests were made
in order to obtain more precise values for the modulus of elasticity at
room and at elevated temperatures. In these additional tests, incre-
ments of stress ranging from 3300 to 4900 psi were repeatedly added to
and removed from the specimens and the resultant strains ranged from 10
to 19 microns. The agreement obtained between the values from these two
types of test and the improved standard deviation for values when using
the larger increments may be seen by comparing the values for body 358
at 1500° and 1800° F given in table 7.

Values for the modulus of elasticity of bodies 358 and 4811C, at
room temperature, were in general higher than the values obtained at
elevated temperatures. Also, bodies 353 and 163 had significantly
lower values of the modulus at 1900° F than at any temperature
between 1500° and 1800° F. The other four bodies showed no significant
difference between the values for the modulus of elasticity at any
elevated temperature.

DISCUSSION

Correlation of Absorption and Extension

No evidence was found of any reduction of diameter in the tension
specimens, even in those specimens where the extreme extension values
were between 2 and 4 percent (for example, test F12-5, table 3). The
decrease in diameter of the specimen along the entire gage length,
necessary to maintain a constant volume, would have been small but
measurable. Since no reduction in diameter, either uniformly along the
gage length or at the point of rupture, was found, there must have been
an increase in the volume of the specimen resulting from the test. It
was noted that those specimens that had been subjected to conditions of
temperature and stress resulting in relatively large amounts of extension
tended to have high absorption values. The suggested hypothesis is that
during the testing, with its accompanying volume increase, the volume of
pores in the body was increased, and an increase in the absorption values
resulted. The extension, which appears to be the most readily available
measure of volume increase, may be correlated with the increase in
absorption.[1] In this correlation, "total extension" was used and it

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[1]Absorption values were obtained by boiling the specimens in carbon
tetrachloride for 5 hours, permitting them to cool in the liquid until
the next day, and determining the increase in weight. The weight increase
was divided by the density of the liquid in order to make the values
comparable with data obtained conventionally by boiling the specimens
in water.

should be borne in mind that this value includes both the elastic deformation, which is recoverable, and the "creep" deformation, most of which is not recoverable.

All specimens were tested for absorption after they had been stressed according to methods 1, 2, 3, or 4. Of all absorption values used in the correlation of absorption and extension, 67 percent were between 0 and 0.10 percent, 27 percent were between 0.10 and 0.50 percent, while 6 percent were over 0.50 percent. The following tabulation gives the maximum extension and absorption for each body:

Body	Maximum extension (percent)	Maximum absorption (percent)	Body	Maximum extension (percent)	Maximum absorption (percent)
358	1.85	0.39	151	2.05	0.94
353	2.00	.71	4811C	.89	.02
163	3.57	.91	16021T	.15	.02

The results for zirconia bodies 358, 353, 163, and 151 showed good rank correlation[2] between percent absorption, determined after testing, and percent total extension. The coefficients of rank correlation obtained for the zirconia bodies in the order given were: +0.82, +0.73, +0.83, and +0.82.

The high-beryllia bodies 4811C and 16021T were characterized by low values for both extension and absorption, regardless of the severity of the testing to which they had been subjected. No significant correlation between absorption and extension was found for these bodies.

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[2]Speerman's formula for rank correlation R, based on unity for perfect correlation, is as follows:

$$R = 1 - \frac{6 \sum d^2}{N(N^2 - 1)}$$

in which

R   coefficient of correlation
d   difference in rank of same specimen with respect to two different characteristics
N   number of specimens

The plus sign before the coefficient indicates that the correlation is positive or direct; that is, the correlated values increase together.

Stress-Temperature-Time Effects

Equalizing strain distributions.- Many failures in the specimen
assembly occurred at locations other than in the gage length. In the
majority of such failures the fracture was in the adapter, the record
showing 23 out of a total of 109 tests on all bodies. The fracture
occurred usually at the inner end of that portion threaded to receive
the specimen (x-x, fig. 1). Of these 23 adapter failures, 12 were
caused by stresses of over 10,000 psi[3] and 16 were caused by stresses
of over 7000 psi.

The supposition that a difference in thermal dilation of adapter
and specimen is responsible for the adapter failure seems untenable.
Bodies 16021T and 4811C have almost identical thermal expansions
(table 2), but only 2 adapters failed during 23 tests of the former
body and 12 adapters failed during 32 tests of the latter. The causes
probably are nonuniform stress distribution in the specimen-adapter
assembly and stress concentrations at the thread roots. Both conditions
are weaknesses inherent in the specimen-adapter assembly. They were
recognized at the time the shapes were designed, but were accepted as
the best compromise in view of the desired precision of the creep
measurements (1 part in 100,000) and the desired uniformity of tempera-
ture ($\pm 5^\circ$ F) over the necessary gage length of 100 millimeters.

That an improved stress distribution can be obtained by some stress-
temperature conditioning well below that required for failure is believed
proved by the results given in table 4. As another illustration,
specimens of body 358 withstood stresses of 15,000, 16,000, and 17,000 psi
after step stressing from 14,000 psi for very long periods of time
(table 5); yet three trials in which stresses of 16,500 or 17,000 psi
were placed initially on specimens all resulted in immediate failure
outside the gage length. Similarly, the specimen of body 151 in
test F1-17 (table 5) withstood a stress of 17,000 psi for 75 hours
before failure in one of the adapters, the specimen remaining intact.
However, five attempts to apply an initial stress ranging from 15,500
to 17,000 psi on other specimens of body 151 all resulted in immediate
failure, of which only one was in the gage length. Furthermore, it is
believed that the apparently lower strength values reported in many tests
for temperatures below 1600° or 1700° F, compared with those for higher
temperatures, are not real but are the result of unequalized stress dis-
tributions. Actual inequalities in strain, from which the bending stress

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[3]The stress values given in the text apply to the gage length of the
specimen. The area of the adapter is approximately 15 times that of the
specimen.

may be calculated as described in reference 25, were measured in a few
tests at points 180° apart. The following examples illustrate the
effect of unequal stressing:

Body	Test	Temperatures at failure (°F)	Stress at failure (psi)	Bending stress (percent)
151	F3-10 (table 3)	1600	19,000	4.2
151	F1-9 (table 3)	1700	13,000	50
151	F8-7 (fig. 12)	1800	18,000	8.2
353	F8-2 (table 3)	1800	12,000	44
353	F1-11 (fig. 8)	1800	18,000	0

Increasing strength.- The evidence presented in tables 5 and 6 is
believed to prove that prolonged temperature testing will cause changes
in the strength and creep-resistant properties of a body. Furthermore,
these changes appear to be beneficial. As a consequence, it cannot be
stated that specimens of a particular composition have a characteristic
strength or creep behavior in tension. On the contrary, the creep rate
for a specified stress-temperature condition may vary several hundred
percent depending on the thermal history of a specimen. This is in
direct contrast to the behavior of a Monel metal investigated by Bennett
and McAdam (see reference 23) who state that their results for this metal,
as a whole, indicate "there was only one characteristic (creep) rate for
each stress-temperature combination."

One direct evidence of an underlying structural change, to account
for the altered strength and creep behavior, is that found in typical
thin sections. It is a fortunate circumstance that a change in structure
can be noted in zirconia bodies such as 358, 353, 163, and 151. The
crystals of zirconia with magnesia in solid solution are normally cubic
and therefore are opaque when viewed with crossed polarized light. In
the stressed and heated specimens, however, these cubic crystals have
partly reverted to the monoclinic form and, consequently, they permit
the passage of some light.

In brief, the results show that (a) the heating and stressing have a
beneficial effect on the porcelains investigated, causing them to be more
resistant to creep and to failure in tension; (b) a structural change is
evidenced by zirconia crystals in specimens subjected to prolonged tests;
and (c) heating without stressing produces the same condition.

Increasing absorption.- So far as the authors know, the phenomenon of a ceramic body elongating permanently under stress with increase in bulk volume, and with commensurate increase in pore volume, has not heretofore been mentioned in the literature. The length-change measurements made in this investigation preclude, in nearly all cases, the accurate determination of the permanent elongation because the elastic recovery was not observed. However, the recovery can be estimated. Calculations for five specimens, representing bodies 358 and 353, show a range in volume increase E in the gage length of 100 millimeters (based on the total elongation corrected for elastic recovery) of from 0.06 to 0.09 cubic centimeters. For the same specimens, the estimated increase in pore volume A (based on absorption determinations) ranged from 0.05 to 0.11 cubic centimeters. Values for E/A ranged from 1.73 to 0.76 and averaged 0.99. It appears probable, therefore, that the increase in bulk volume of the specimens during the creep tests is accompanied by a proportionate increase in pore volume. The nature of this pore space was not determined and was not detected in the thin sections examined microscopically.

General Evaluation of Bodies

In exploratory tests, such as conducted in this study for the selection of bodies to be used in creep tests, it is believed that the bending test serves a useful purpose. It will cull out the definitely weak bodies (as illustrated in table 1 of reference 13); however, in this study at least, it did not place the stronger bodies in the same order as the tension test did. As an illustration, the bending test showed body 4811C considerably inferior to body 353 at 1800° F. The reverse was indicated by the tension tests. A variable other than composition is involved, however. The bending tests were conducted on bars made in the laboratory, and the tension specimens were fabricated by an industrial concern. Two bars of body 353 and two bars of body 4811C, made by the industrial concern with the same procedure used to fabricate the tension test specimens, were broken in bending at 1800° F. The bars of body 353 were somewhat weaker than those made in the laboratory, and the bars of body 4811C were about 50 percent stronger. As a result, these four tests showed body 4811C to be stronger than body 353. The inference is that the bending test and the tension test may place compositions in the same order of relative strength if the specimens for both are fabricated in the same way. This emphasizes the importance of conducting tests on specimens made by the same process to be used in making the actual physical shapes destined for service.

It is obvious that the density of a ceramic body to be used in
applications such as turbine blades must be considered when comparisons
are made with other ceramics or with metals. The figures of merit given
in table 3 are admittedly of limited significance because a comprehensive
figure of merit would include the factors of creep, thermal dilation,
thermal conductivity, and also elasticity. The importance of density is
shown by comparing body 4811C with a density of 3 grams per cubic centi-
meter and Vitallium with a typical density of 8.32 grams per cubic centi-
meter. Thus, a strength in tension of 18,000 psi at 1800° F for the
ceramic body would be equivalent to a strength of 50,000 psi for the
metal.

National Bureau of Standards
      Washington 25, D. C., Oct. 6, 1947

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TABLE 1.- VAPOR TEST

Body designation	Absorption (percent)	Increase in weight (percent)	Modulus of rupture (psi) at room temperature -	
			Following the vapor test	Untreated (a)
Commercial bodies				
2673	1.8	0	6,000	31,400
2673	2.3	0	8,000	
3239	0	0	14,200	19,200
b3239				
NBS bodies				
4811C	0.13	0.01	26,900	25,200
4811C	.01	.01	22,400	
16021T	.04	.01	19,700	20,200
16021T	.95	.02	19,500	
353	0	.01	33,200	26,200
353	0	.01	35,400	
358	.01	0	19,000	25,000
358	.01	0	15,200	

aRepresentative value for untreated bars.
bBroke in furnace during vapor treatment.

TABLE 2.— COMPOSITION AND PROPERTIES OF TENSION TEST SPECIMENS

Body designation (1)	Composition, by weight (percent)							Bulk density (grams/cc)	Linear thermal expansion (percent) (2)
	MgO	CaO	BeO	Al_2O_3	ThO_2	ZrO_2	TiO_2		
358	9.8	---	10.2	---	---	80.0	---	4.9	1.36
353	19.6	---	20.3	---	---	60.1	---	4.4	1.31
163	7.2	---	26.8	---	---	66.0	---	4.4	1.23
151	14.0	---	43.3	---	---	42.7	---	3.8	1.16
16021T	----	---	90.0	4.3	5.7	----	2.0	3.0	1.11
4811C	----	2.0	84.2	7.2	---	8.6	---	3.0	1.10

[1]358 $3MgO:5BeO:8ZrO_2$ (mole).
    353 . $3MgO:5BeO:3ZrO_2$ (mole).
    163      $MgO:6BeO:3ZrO_2$ (mole).
    .151 $MgO:5BeO:ZrO_2$ (mole).
    16021T $160BeO:2Al_2O_3:ThO_2$ (mole) plus 2 percent TiO_2 by weight.
    4811C $48BeO:Al_2O_3:ZrO_2$ (mole) plus 2 percent CaO by weight.

[2]For the range, room temperature to 1200° C.
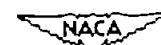
TABLE 3.— SUMMARY OF RESULTS FROM STEP TESTS BY METHODS 1 AND 2

[Tests are grouped by temperature at which failure occurred
and only those tests are summarized in which failure
came in the constricted portion of the specimen]

Body designation	Test	Total duration of test (hr)	Total extension (percent) (a)	Maximum observed rate of creep (percent/hr)	Duration of test temperature (hr)	Stress at failure (psi)	Figure of merit, $\frac{\text{Stress at failure}}{\text{Density}}$
Room Temperature							
358	F4-3	836	0.027	Negligible	0.5	9,000	18×10^2
151	F4-5	245	.028	----do----	2.0	8,000	21
16021T	F4-4	1252	.024	----do----	151.0	11,000	37
1500° F							
358	F3-6	1104	0.006	0.35×10^{-4}	0.14	13,000	27
353	F10-1	1554	.029	.26	1.0	13,000	30
151	F6-3	900	.027	Negligible	2.5	12,000	32
4811C	F1-7	1627	.021	.39	.25	15,000	50
1600° F							
358	F2-8	1543	.0.035	0.26	40.0	13,000	27
151	F3-10	2538	b.103	.31	102.5	19,000	50
16021T	F7-8	1647	.046	.37	48.0	13,000	43
4811C	F7-7	2833	.145	1.00	1.5	20,000	67
1700° F							
358	F8-3	1268	0.103	2.08	91.5	14,000	29
358	F1-13	2063	.195	1.01	51.0	16,000	33
353	F2-7	2734	.242	1.69	83.0	19,000	43
163	F10-4	2876	.284	1.57	104.0	20,000	45
151	F1-9	1560	.008	Negligible	103.5	13,000	34
151	F2-11	1535	.105	.82	24.5	13,000	34
16021T	F8-6	978	.101	1.29	1.5	10,000	33
16021T	F6-4	1406	.152	2.51	44.5	13,000	43
4811C	F9-8	1908	.150	.99	114.0	14,000	47
4811C	F9-9	1995	.148	.49	10.0	14,000	47
1800° F							
358	F11-6	4438	'1.853	c16.0	d2.0	18,000	37
353	F8-2	2141	.184	1.30	8.5	12,000	27
353	F1-11	2855	b1.345	14.75	81.5	18,000	41
353	F12-1	1591	.510	c18.2	e141.0	7,000	16
163	F7-6	2184	.785	6.69	6.0	16,000	36
151	F8-7	2347	.492	4.23	6.5	18,000	47
16021T	F2-9	347	.092	2.18	36.5	6,000	20
16021T	F9-13	359	.030·	.59	22.5	6,000	20
16021T	F1-10	379	.064	2.0	67.0	6,000	20
16021T	F1-8	929	.047	9.53	39.5	7,000	23
4811C	F8-5	2351	.381	3.0	3.5	18,000	60
1900° F							
358	F11-5	3385	b1.735	23.2	83.5	8,000	16
353	F7-9	698	.164	4.72	2.5	6,000	14
353	F12-8	1696	1.98	34.1	25.0	11,000	25
163	F10-6	1505	b1.827	32.1	57.5	11,000	25
151	F7-10	2022	1.345	22.9	37.5	15,000	39
16021T	F12-3	796	.030	.73	5.0	4,000	13
4811C	F11-7	921	.634	16.2	108.5	16,000	53
1950° F							
358	F12-7	1996	1.547	41.4	275.5	6,000	12
2000° F							
163	F12-5	1963	3.566	f69.9	309.0	4,000	9
151	F12-2	1822	b1.317	f60.0	153.5	4,000	11
2100° F							
4811C	F10-7	1789	0.859	12.7	71.0	6,000	20
2230° F							
4811C	F11-2	2094	0.339	g24.1	0.75	4,000	13

a Includes the elastic deformation.

b This value includes some strain which was recovered, but not measured, when the specimen was unloaded preliminary to installing new gages or repairing furnace.

c Observed while specimen was held at 1950° F.

d Specimen had been held at 1950° F for 457 hr prior to holding at 1800° F.

e Specimen had been held at 1950° F for 96 hr prior to holding at 1800° F.

f Observed while specimen was held at 2050° F.

g Observed while specimen was held at 2100° F.
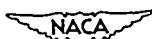
TABLE 4.- SUMMARY OF SHORT-TIME TENSILE TESTS AT 1800° F

[Specimens reported in group A had no previous stressing;
those reported in group B were prestressed for 48 hr
at 1800° F and 4000 psi in addition to previous tests,
if any, referred to under Remarks]

Body designation	Test	Stress at rupture (psi)	Location of fracture	Remarks
		Group A		
163	F9-5	6,400	Adapter	-----
163	F3-7	9,980	----do-----	-----
163	F7-5	5,870	----do-----	-----
4811C	F9-4	18,990	----do-----	-----
4811C	F9-6	18,370	----do-----	-----
4811C	F9-7	4,610	----do-----	-----
4811C	F7-3	11,750	----do-----	-----
4811C	F7-4	10,060	Gage length	-----
		Group B		
358	F9-16	14,510	Gage length	No previous test
358	F7-15	13,980	----do-----	Do.
151	F7-12	19,120	----do-----	For previous test see fig. 15
151	F8-9	16,650	----do-----	For previous test see fig. 14
151	F9-15	17,830	----do-----	No previous test
16021T	F8-11	[a]4,000	----do-----	Do.
16021T	F8-12	9,880	----do-----	Do.
16021T	F8-13	[a]4,000	----do-----	Do.
4811C	F7-16	19,270	----do-----	Do.
4811C	F8-14	15,800	----do-----	(b)
4811C	F9-18	10,320	Adapter	No previous test

[a]Failure occurred during the prestressing at 4000 psi and 1800° F.

[b]This specimen had been tested for 2125 hr under 4000-psi stress at temperatures ranging from 1600° to 2100° F. The results were not reported because the gages were found to be defective.

27
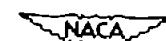
TABLE 5.- CREEP RATES OBSERVED AT 1800° F DURING STRESS-RUPTURE TESTS

Body designation	Test	Stress (psi)	Duration of stressing (hr)	Average creep rate (percent/hr) for the period -					
				1 to 100 hr	100 to 200 hr	200 to 500 hr	500 to 1000 hr	1000 to 1500 hr	1500 to 2000 hr
358	F2-13	10,000	2040	6.51×10^{-4}	4.96×10^{-4}	4.84×10^{-4}	3.20×10^{-4}	2.34×10^{-4}	1.84×10^{-4}
		12,000	623	3.94	2.71	2.83	------------		
		14,000	748	5.02	3.72	3.94	(a)	------------	------------
358	F7-17	15,000	2631	13.8	10.6	10.6	7.03	5.62	5.40
358	F3-12	14,000	1627	8.36	6.15	5.92	4.45	3.16	------------
		15,500	96	3.38	------------	------------	------------	------------	------------
		16,000	601	3.83	3.97	[b]3.60	------------	------------	------------
		17,000	500	4.96	4.66	[b]4.42	------------	------------	------------
358	F12-14	16,000	1373	14.9	11.7	11.5	8.39	------------	------------
163	F3-9	5,000	936	2.21	.67	.57	------------	------------	------------
		6,000	1197	2.21	1.22	.80	1.09	------------	------------
		8,000	888	1.98	1.88	1.24	------------	------------	------------
		10,000	1200	4.20	2.46	2.21	1.99	------------	------------
		12,000	1203	3.89	3.51	3.24	3.22	------------	------------
		14,000	1907	8.55	4.60	4.17	4.33	------------	------------
		16,000	621	7.50	7.58	7.70	(c)	------------	------------
151	F1-17	12,000	1001	2.55	2.06	2.02	1.64	------------	------------
		16,000	504	3.98	3.61	2.84	------------	------------	------------
		[d]16,000	503	8.15	4.67	3.61	------------	------------	------------
		17,000	75	(a)	------------	------------	------------	------------	------------
1602T	F9-19	7,000	1/4	(c)	------------	------------	------------	------------	------------
1602T	F2-10	6,000	3/4	(c)	------------	------------	------------	------------	------------
1602T	F1-14	5,000	4	(c)	------------	------------	------------	------------	------------
1602T	F7-11	6,000	283	4.13	1.93	(c)	------------	------------	------------
1602T	F1-16	4,000	995	3.56	1.46	1.02	.40	(b)	------------
48110	F8-8	8,000	1001	2.23	.67	.67	.45	------------	------------
		12,000	555	1.95	1.53	1.12	------------	------------	------------
		16,000	503	5.24	2.83	2.26	------------	------------	------------
		20,000	1/60	(a)	------------	------------	------------	------------	------------
48110	F6-6	9,000	22	------------	------------	------------	------------	------------	------------
		12,000	9	(e)	------------	------------	------------	------------	------------

[a]Adapter failed.

[b]Test was discontinued.

[c]Failed in gage length.

[d]The specimen was reloaded at 16,000 psi, after 143 hr at 1800° F and no load, for recovery of strain.

[e]Specimen failed in fillet.
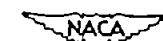
TABLE 6.- TEMPERATURE-STRESS-TIME EFFECTS ON CREEP RATE

Body designation	For data see -	Prior testing conditions	Test conditions maintained while average creep rate was obtained			
			Temperature (°F)	Stress (psi)	Duration (hr)	Average creep rate (percent/hr)
(1)	(2)	(3)	(4)	(5)	(6)	(7)
358	Table 5	a No prior testing	1800	15,000	430	11.3×10^{-4}
	Fig. 4	b 1800° F; 4,000 to 15,000 psi	1800	15,000	430	5.45
358	Table 5	a No prior testing	1800	14,000	792	5.96
	Table 5	b 1800°; 10,000 to 14,000 psi	1800	14,000	738	3.98
358	Table 3	a 1600° to 1950° F; 4,000 psi	1950	4,000	269	16.0
	Table 3	b 1950° to 1700° to 1950° F; 4,000 psi	1950	4,000	168	13.6
358	Table 3	a 1600° to 1800° F; 4,000 psi	1800	4,000	166	2.18
	Table 3	b 1800° to 1950° to 1700° to 1800° F; 4,000 psi	1800	4,000	172	.54
358	Fig. 7	a 1600° to 1950° F; 6,000 psi	1950	6,000	167	41.4
	Fig. 7	b 1950° to 1700° to 1950° F; 6,000 psi	1950	6,000	95	27.6
353	Table 3	a 1350° to 1800° F; 4,000 psi	1800	4,000	144	2.52
	Table 3	b 1800° to 1950° to 1800° F; 4,000 psi	1800	4,000	122	.28
151	Table 5	a No prior testing	1800	12,000	286	2.46
	Table 3	b 1800° F; 4,000 to 12,000 psi	1800	12,000	174	1.32
48110	Table 5	a No prior testing	1800	8,000	185	1.50
	Fig. 18	b 1800° F; 4,000 to 8,000 psi	1800	8,000	169	.79
48110	Fig. 20	a 1600° to 2000° F; 6,000 psi	2000	6,000	166	5.81
	Fig. 20	b 2000° to 2100° to 2000° F; 6,000 psi	2000	6,000	163	3.60
48110	Fig. 20	a 1600° to 2050° F; 6,000 psi	2050	6,000	168	12.7
	Fig. 20	b 2050° to 2000° to 2050° F; 6,000 psi	2050	6,000	168	11.0

a Range of temperature and stress used during first stage of testing prior to test conditions given in columns (4), (5), and (6).

b Range of temperature and stress used subsequent to the first stage and test conditions described in footnote a and prior to test conditions given in columns (4), (5), and (6).

TABLE 7.- MODULUS OF ELASTICITY AT VARIOUS TEMPERATURES

[Unless noted otherwise, the values of the modulus are based on data from step tests in which the stress increments usually were 1000 psi]

Body designation	Number of values	Temperature (°F)	Mean modulus of elasticity (psi) (a)	Body designation	Number of values	Temperature (°F)	Mean modulus of elasticity (psi) (a)
358	18	Room	b$(32 \pm 3.0) \times 10^6$	151	7	1500	$(32 \pm 15) \times 10^6$
	10	1500	b(20 ± 1.6)		16	1600	(31 ± 8.2)
	10	1500	(35 ± 13)		16	1700	(37 ± 16)
	12	1600	(24 ± 12)		18	1800	(25 ± 8.4)
	20	1700	(29 ± 10)		13	1900	(33 ± 22)
	10	1800	b(22 ± 1.0)		70	1500 to 1900	c(31 ± 15)
	36	1800	(23 ± 13)	16021T	12	1500	(32 ± 19)
	10	1900	(21 ± 5.6)		8	1600	(37 ± 16)
	88	1500 to 1900	c(26 ± 12)		9	1700	(35 ± 15)
353	15	1500	(28 ± 12)		9	1800	(30 ± 17)
	7	1600	(43 ± 23)		38	1500 to 1800	c(33 ± 17)
	24	1700	(32 ± 12)	4811C	8	Room	b(44 ± 2.9)
	28	1800	(26 ± 11)		14	1500	(37 ± 15)
	12	1900	(15 ± 5.3)		15	1600	(48 ± 23)
	74	1500 to 1800	c(30 ± 19)		18	1700	(32 ± 15)
163	9	1500	(32 ± 12)		28	1800	(36 ± 19)
	32	1700	(24 ± 8)		18	1900	(32 ± 17)
	22	1800	(25 ± 8)		95	1500 to 1900	c(36 ± 19)
	10	1900	(25 ± 5)				
	64	1500 to 1800	c(26 ± 9.2)				

[a]The second part of the expression for modulus of elasticity is the standard deviation.

[b]This value is based on a test in which stresses ranging from 3300 psi to 4900 psi were repeatedly added to and removed from the specimen.

[c]This value is based on all results for that range of temperature within which there is no significant difference between mean values obtained at even 100° intervals but not including the results obtained as described in footnote b.

Silicon-carbide cement

Two gages used on each specimen 180° apart

100 mm

B

A

Platinum tube

Platinum wire

SECTION ON A-A

ASSEMBLED ADAPTERS, SPECIMEN
AND GAGE

B

Approximate size of extensometer field

Reference lines scored on polished tube and wire

A   A

Platinum tube

Platinum wire

A

EXTENSOMETER GAGE
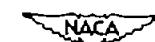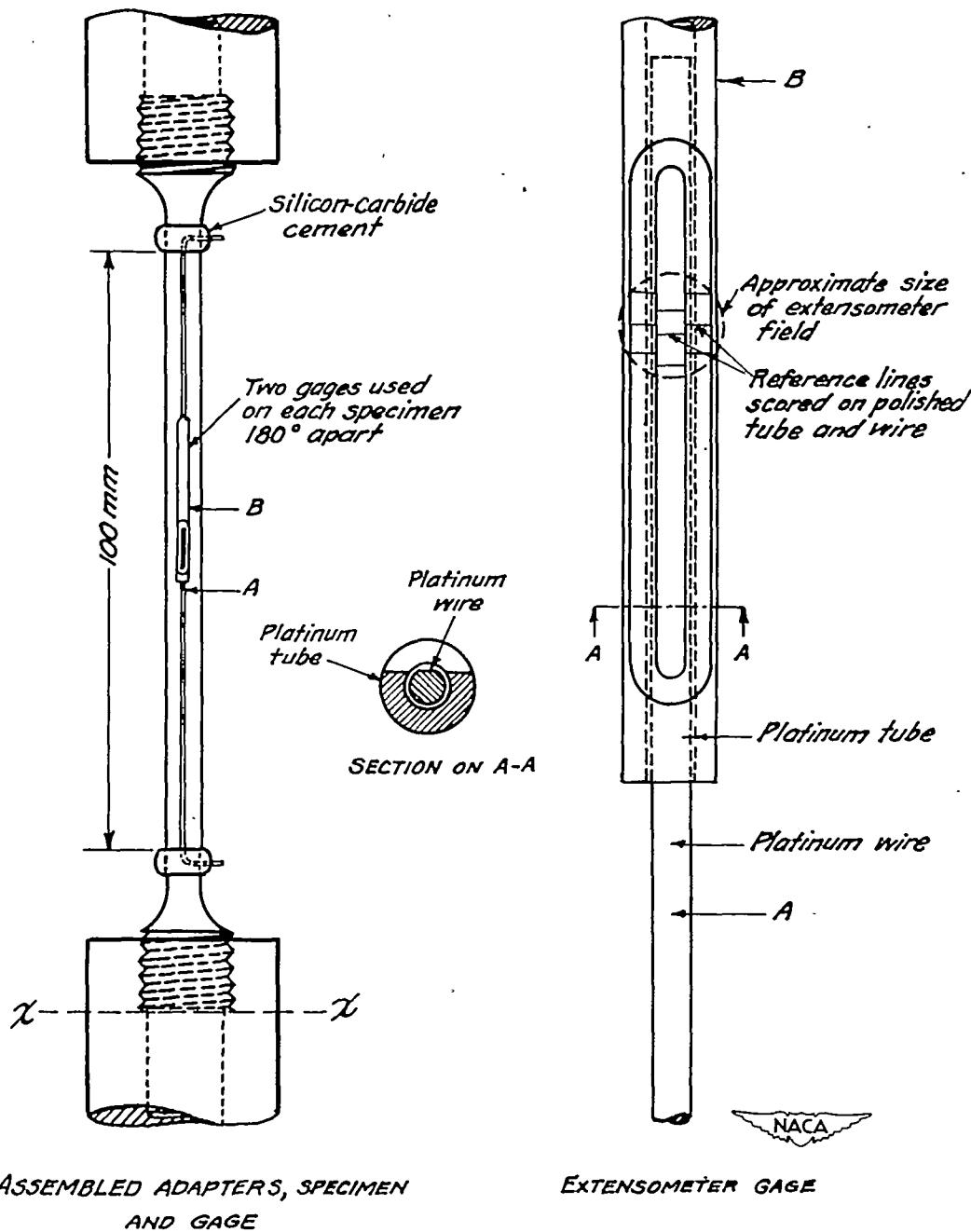
Figure 1.- Assembly of tension specimen, with ceramic adapters and gage
for observing length changes, and an enlarged view of the strain gage.
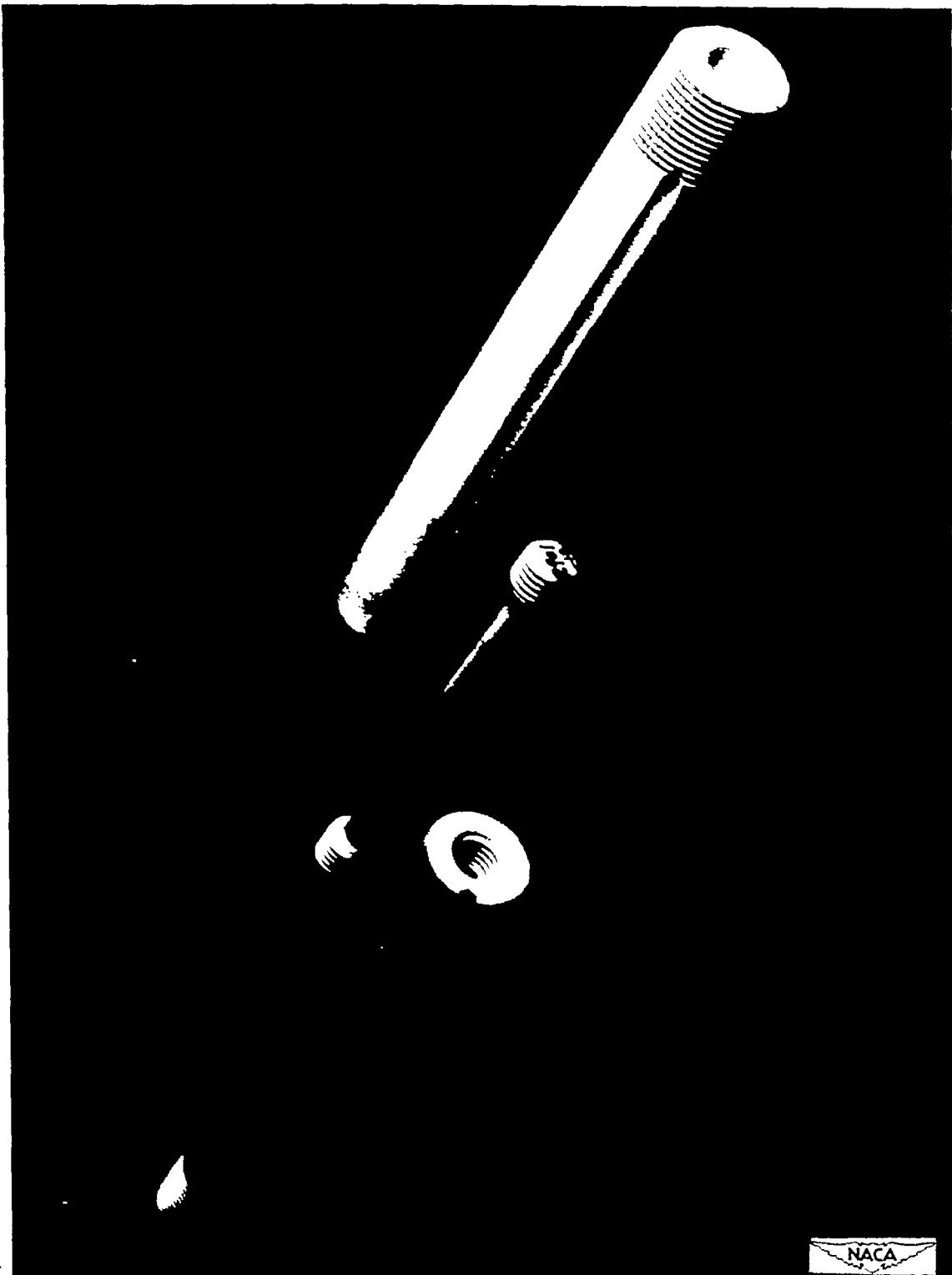
Figure 2.- Threaded tensile specimen and two ceramic adapters.
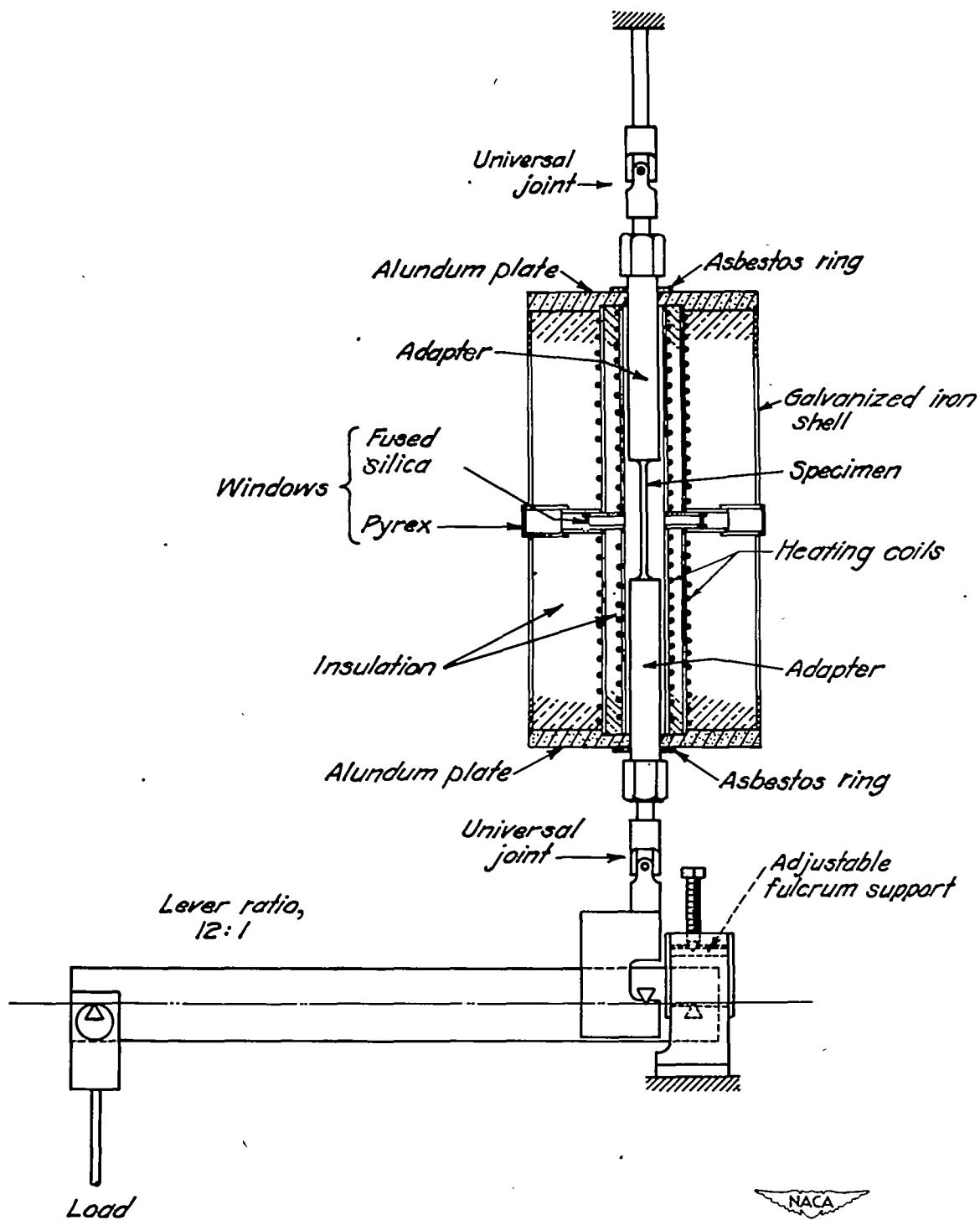
Figure 3.- Tension-specimen assembly in furnace and the loading system.
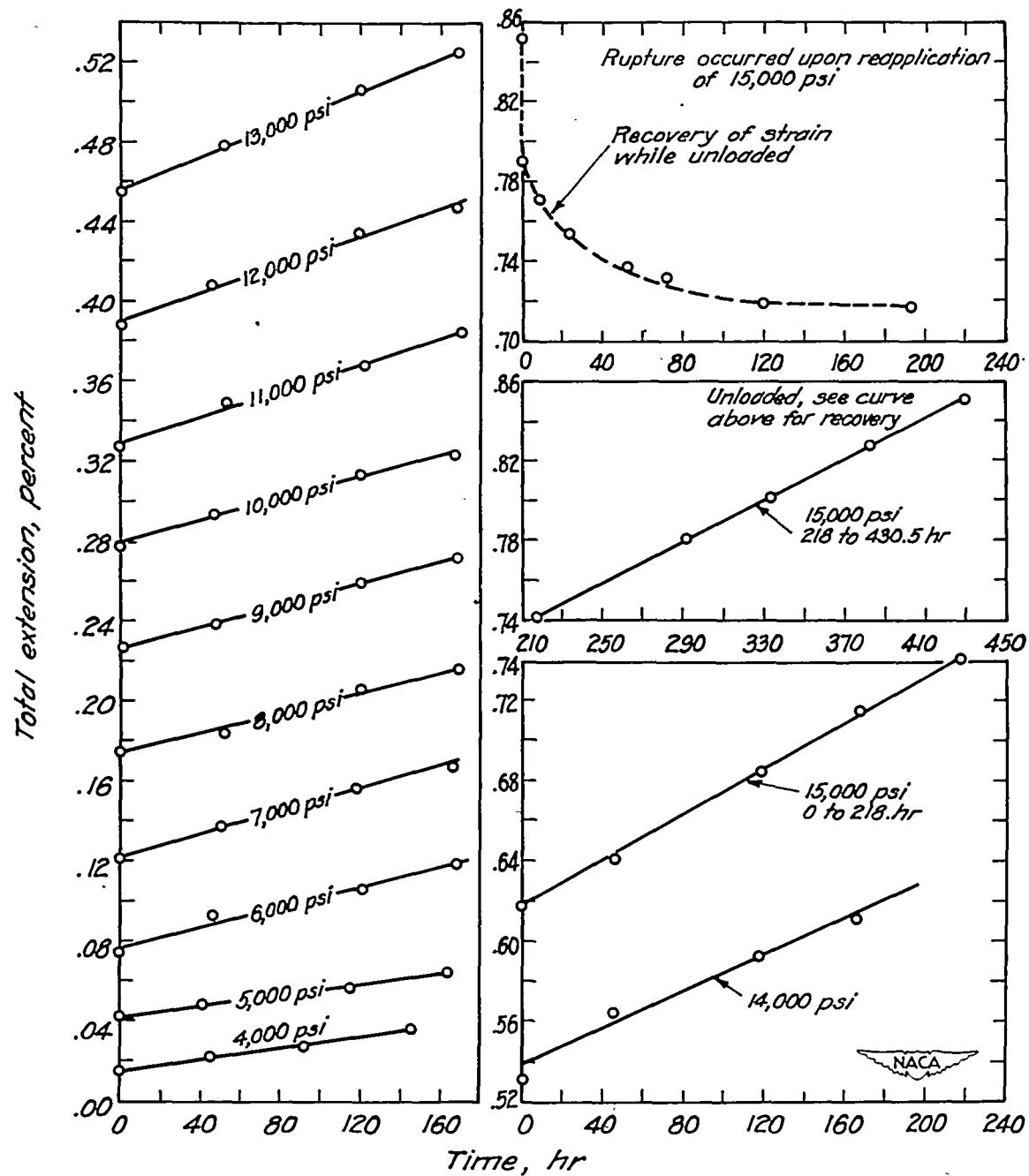
Figure 4.- Test F8-4. Time-extension data for body 358. This specimen
was tested at 1800° F at various stresses until failure. At the end of
430.5 hours at 1800° F and 15,000 psi, the load was removed because
the power was shut off for 2 hours. However, recovery was observed ·
for 193 hours. Failure occurred in the threaded portion of the specimen.
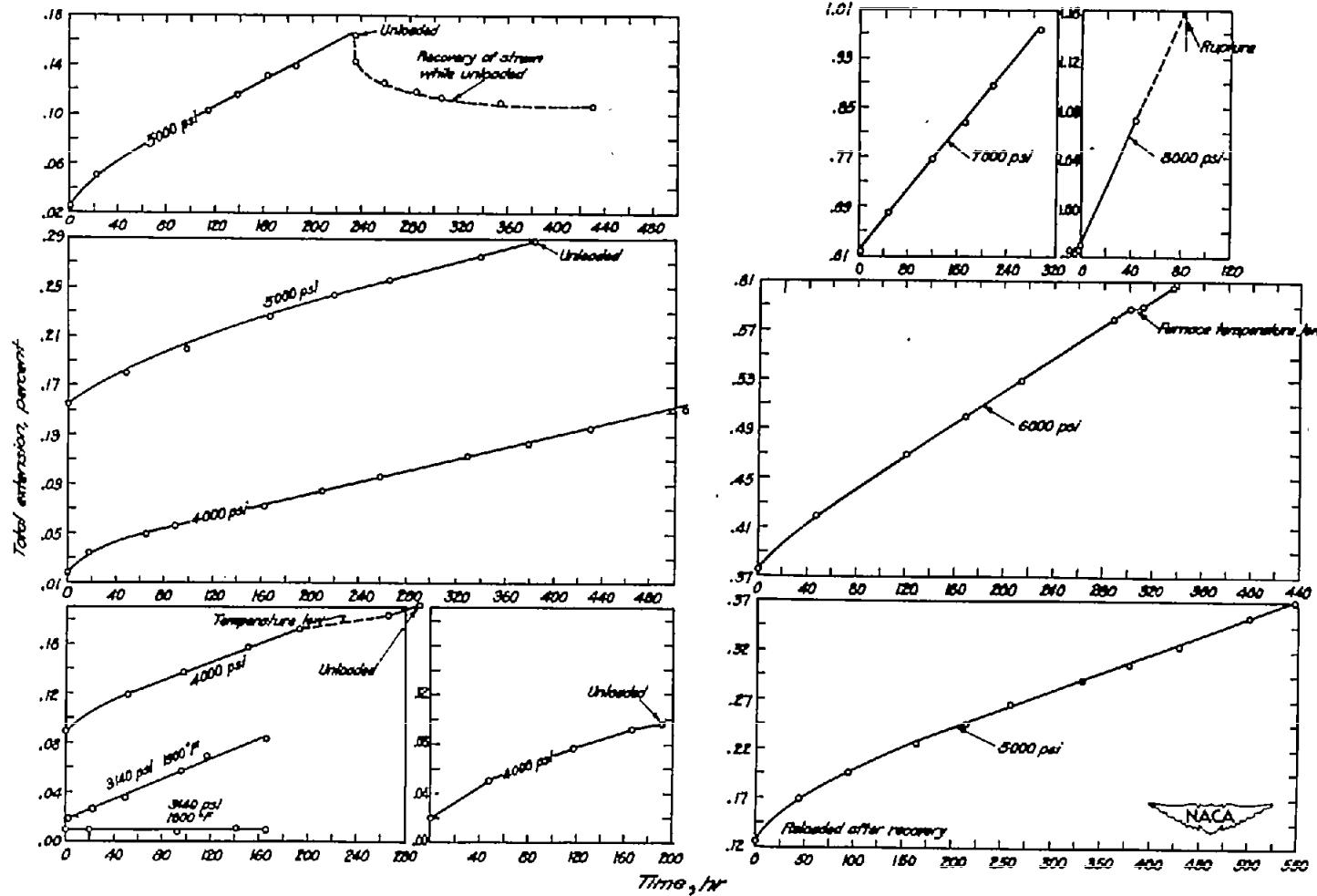
Figure 5.- Test F11-5. Time-extension data for body 358. This specimen was tested at 1900° F at various stresses until failure. Measurements of the diameter before testing and after rupture showed no evidence of reduction of area as a result of over 1 percent of extension. Failure occurred near the center of the gage length.
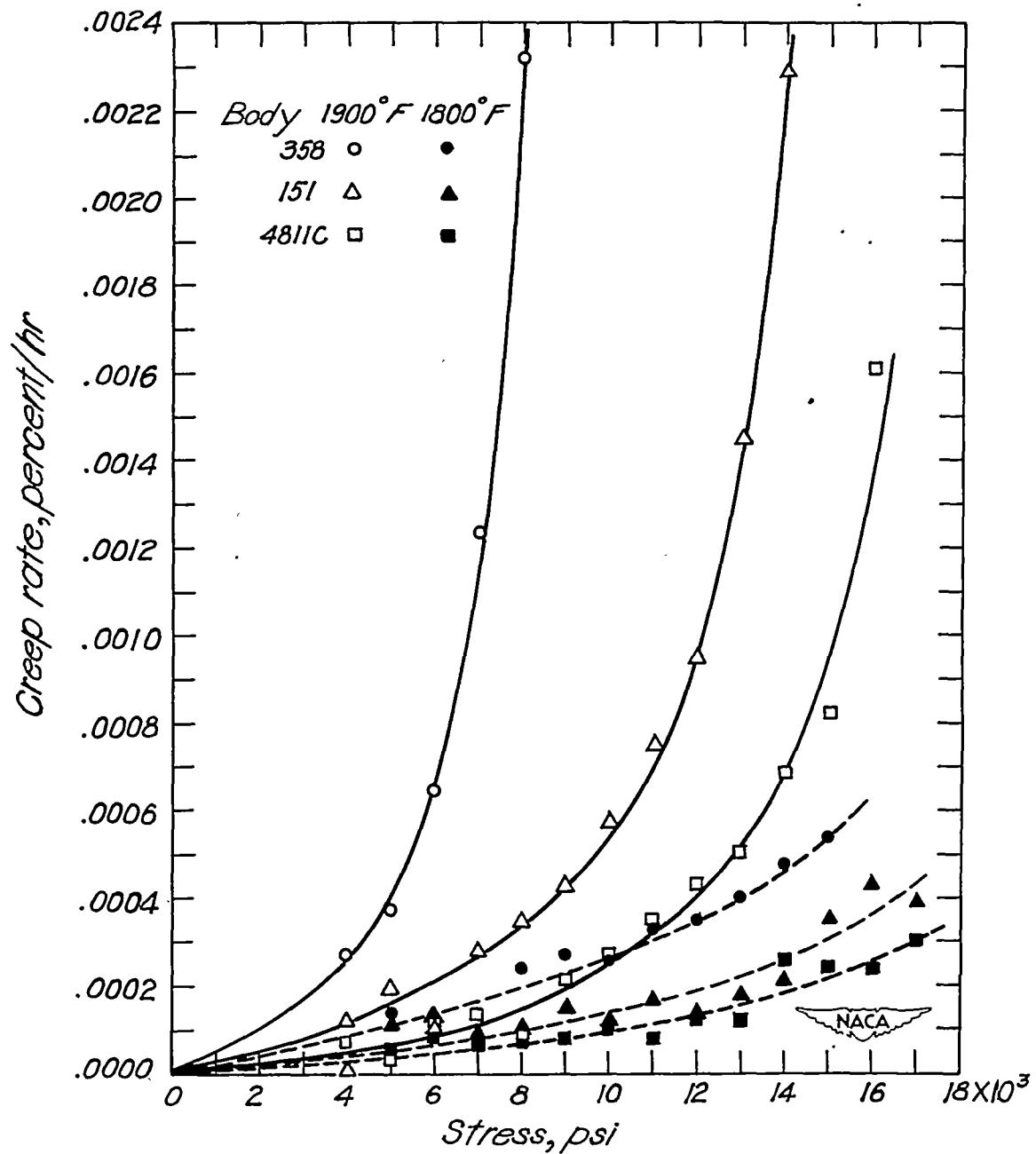
Figure 6.-   Typical curves of creep rate against stress at 1800° and 1900° F
for bodies 358, 151, and 4811C.
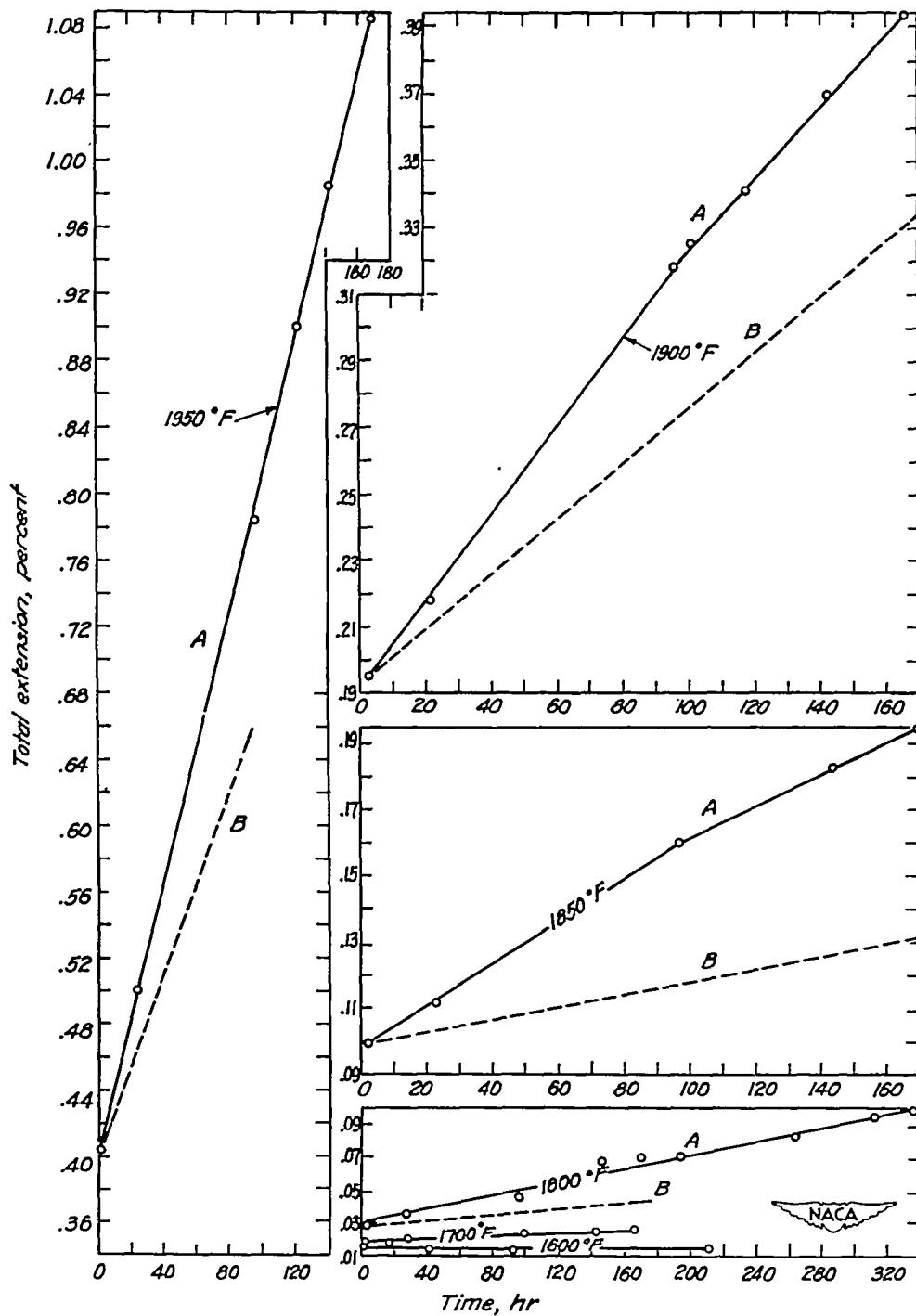
Figure 7.- Test F12-7. Time-extension data for body 358. This specimen was tested at a constant stress of 6000 psi. In the first part of the test (curves A), the temperature was increased by increments from 1600° to 1950° F and then dropped to 1700° F. In the second part (curves B), the temperature was increased from 1700° to 1950° F. Failure occurred in the gage length.
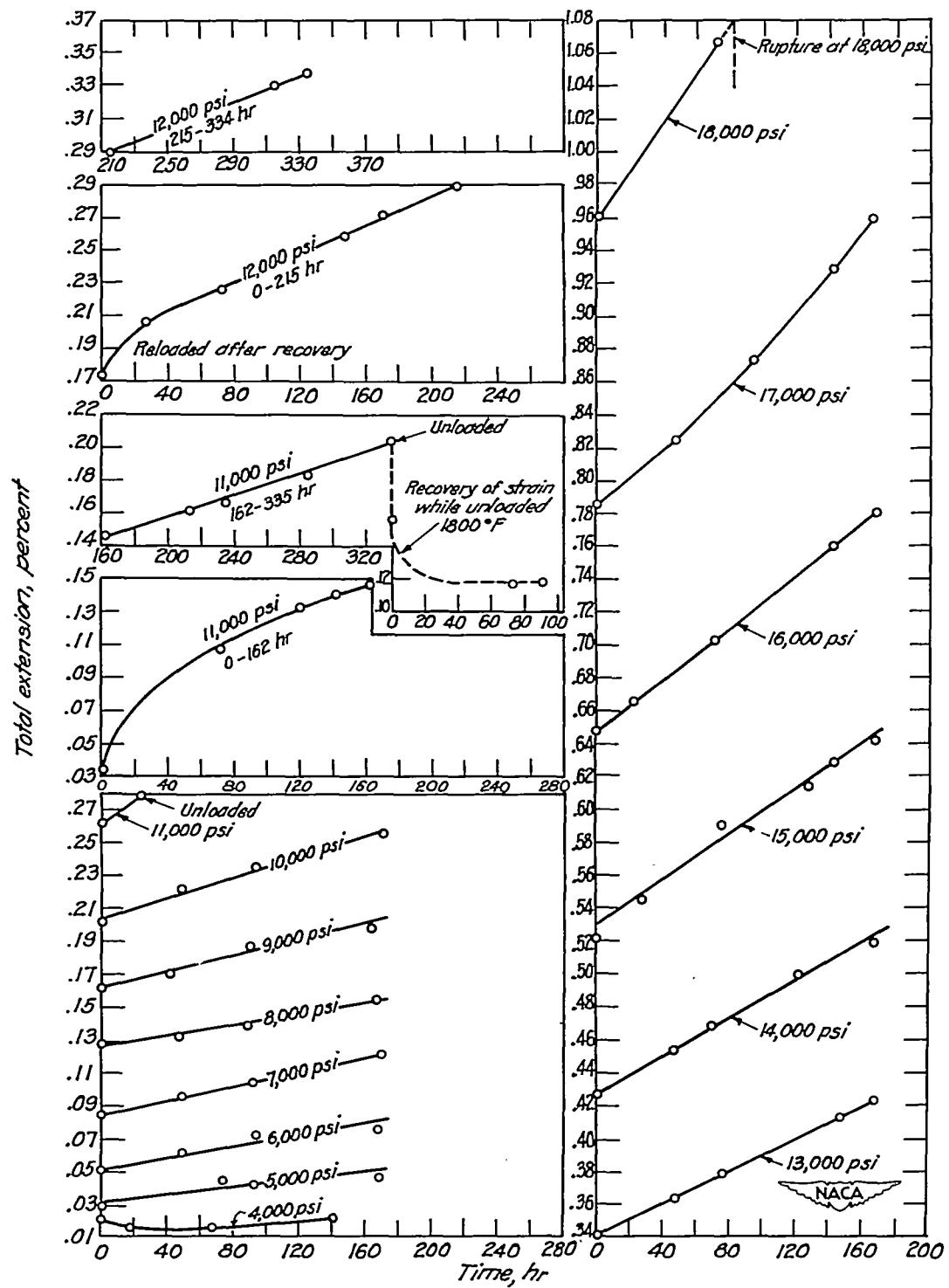
Figure 8.- Test F1-11. Time-extension data for body 353. This specimen
was tested at 1800° F at various stresses until failure which occurred
near the top fillet at the same time that a specimen failed in an adjacent
furnace.
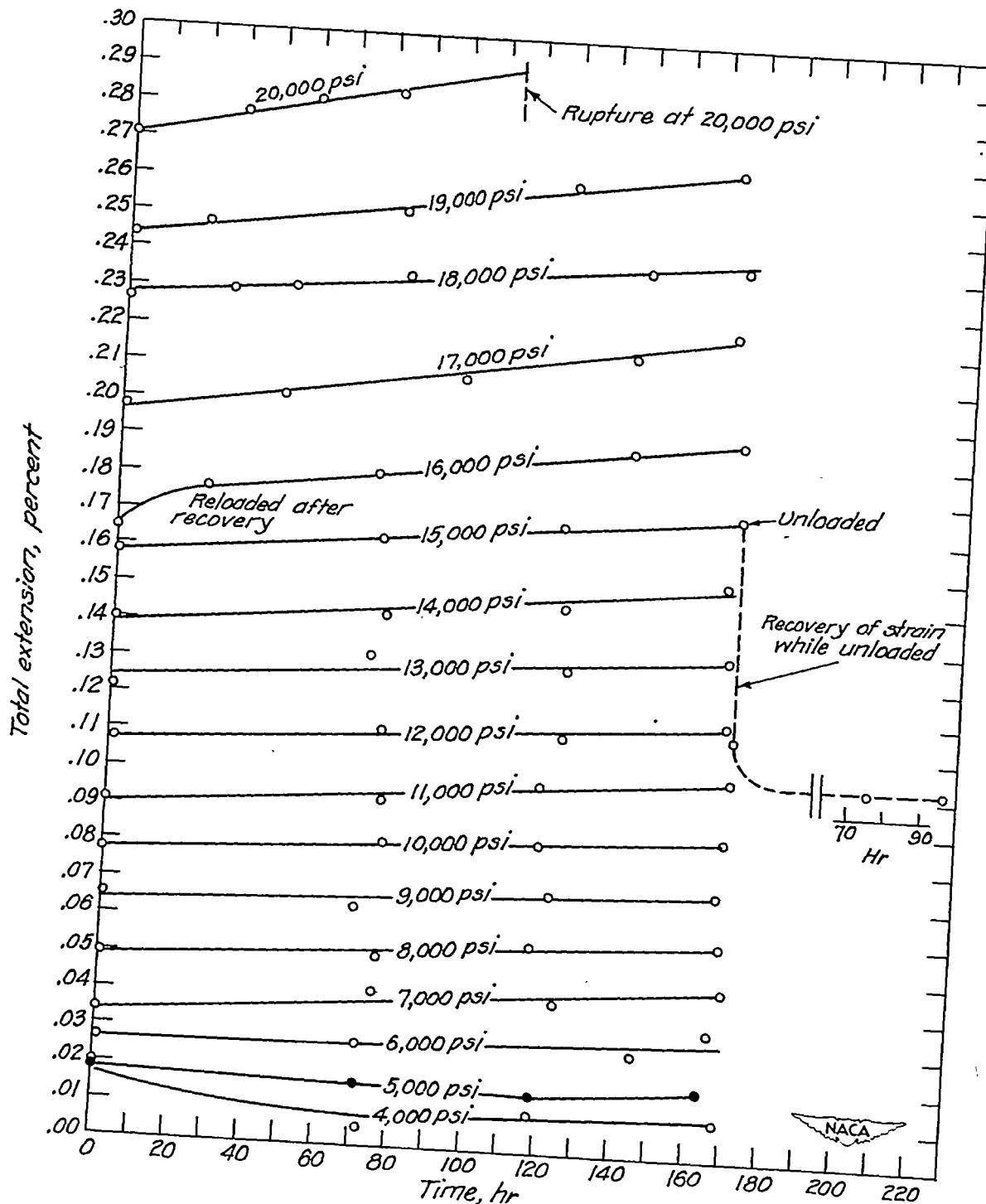
Figure 9.- Test F10-4. Time-extension data for body 163. This specimen was tested at 1700° F at various stresses until failure. Failure occurred near the fillet.
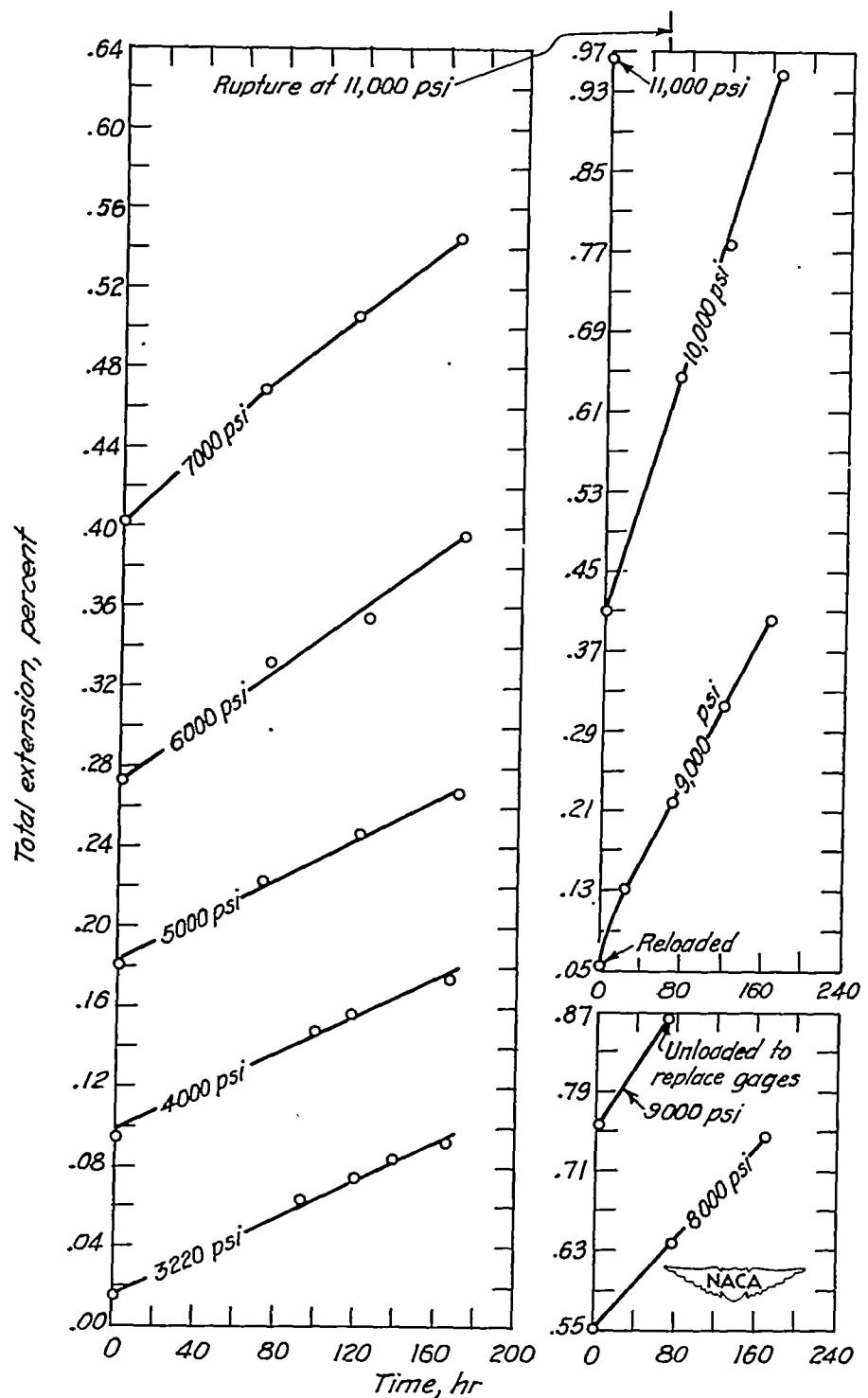
Figure 10.- Test F10-6. Time-extension data for body 163. This specimen was tested at a constant temperature of 1900° F at various stresses until failure. Rupture occurred within the gage length.
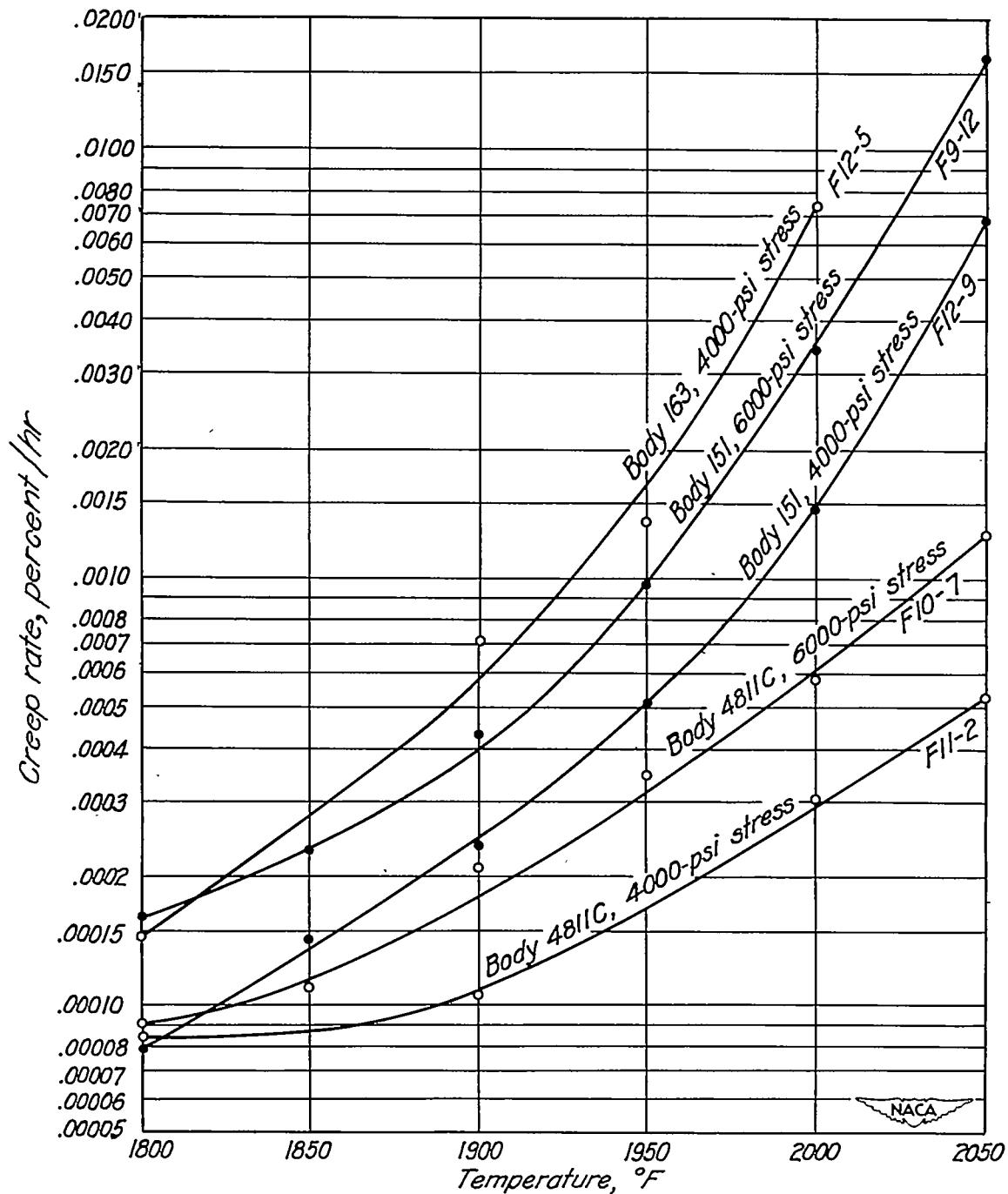
Figure 11.- Curves of creep rate against temperature at constant stress for bodies 163, 151, and 4811C.
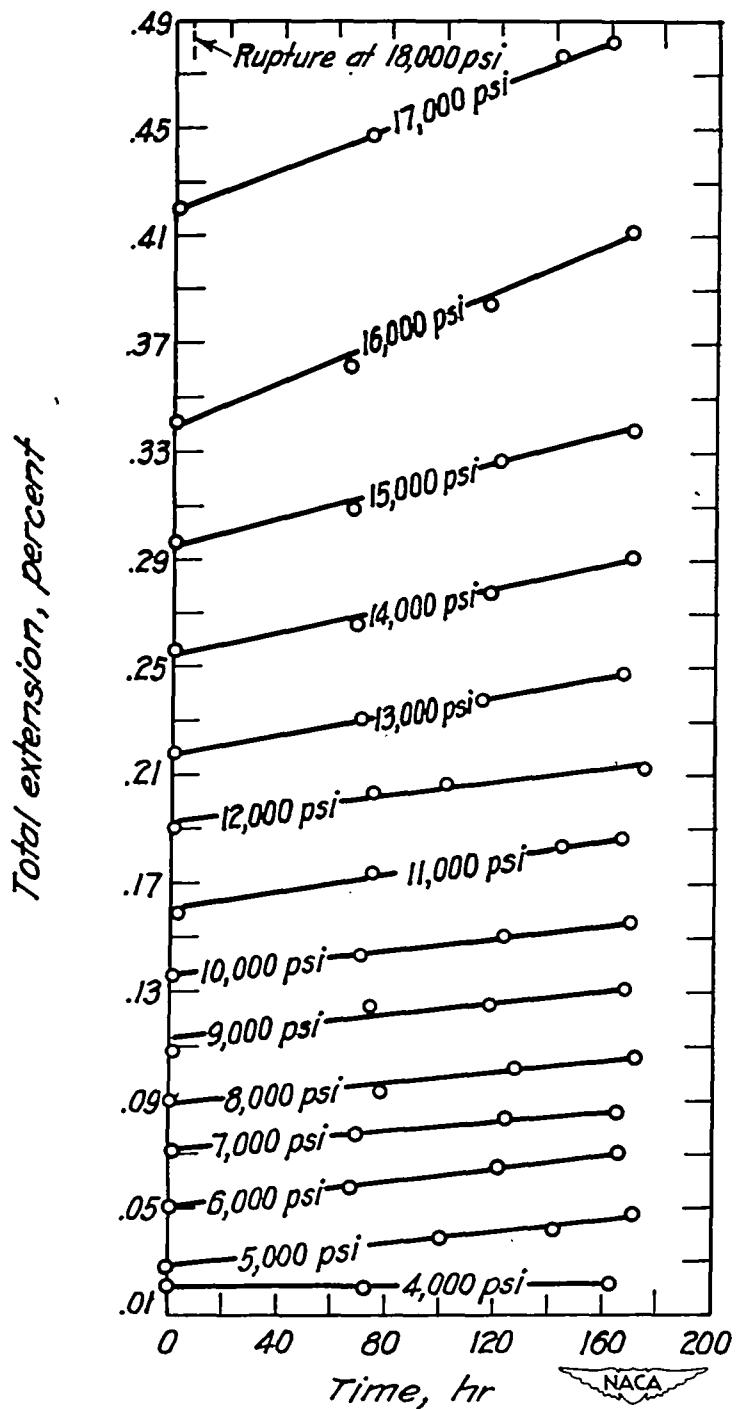
Figure 12.- Test F8-7. Time-extension data for body 151. This specimen
was tested at a constant temperature of 1800° F at various stresses until
failure. Rupture occurred near the bottom fillet.
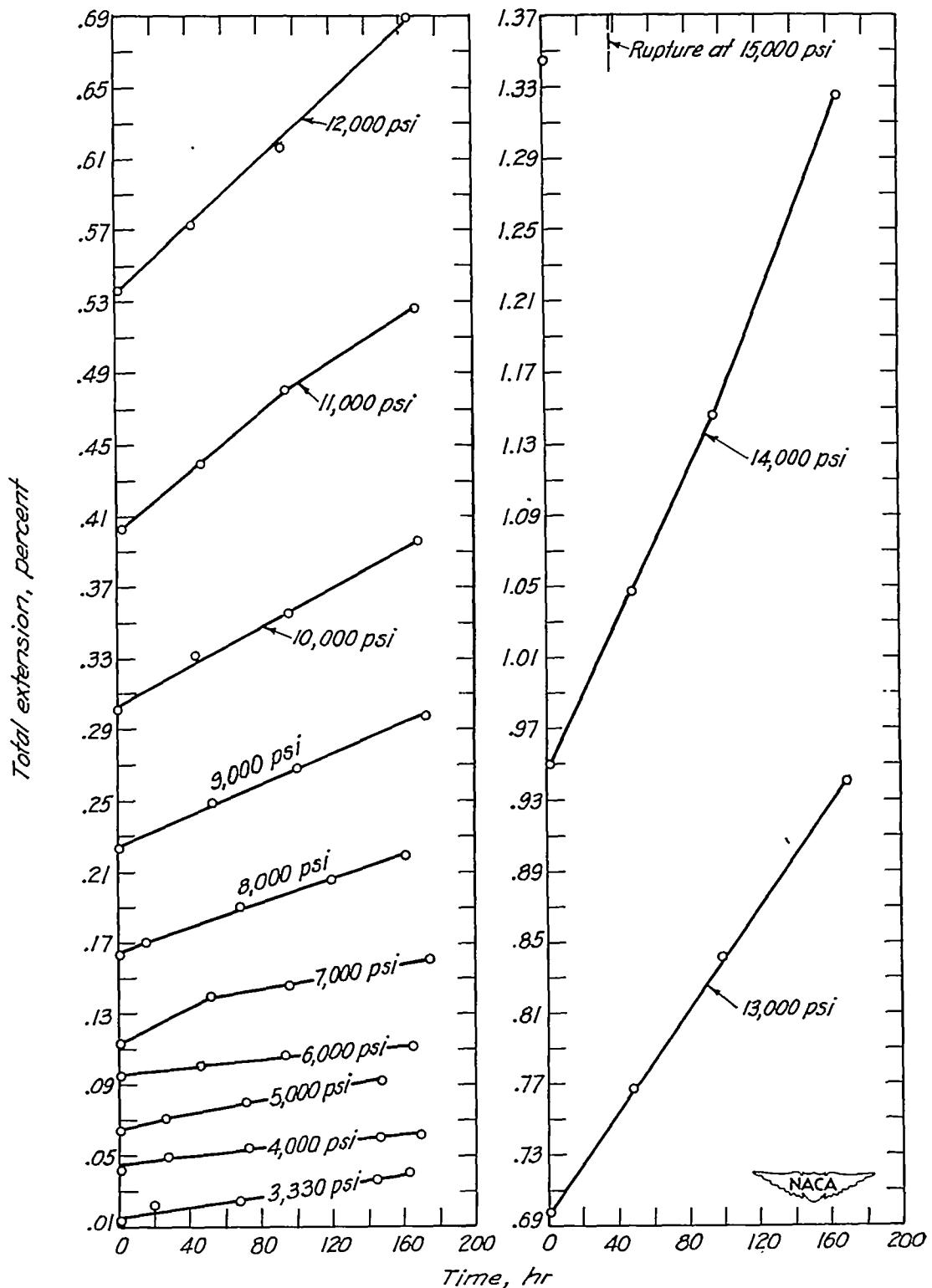
Figure 13.- Test F7-10. Time-extension data for body 151. This specimen
   was tested at a constant temperature of 1900° F at various stresses until
   failure. Rupture occurred in the gage length.
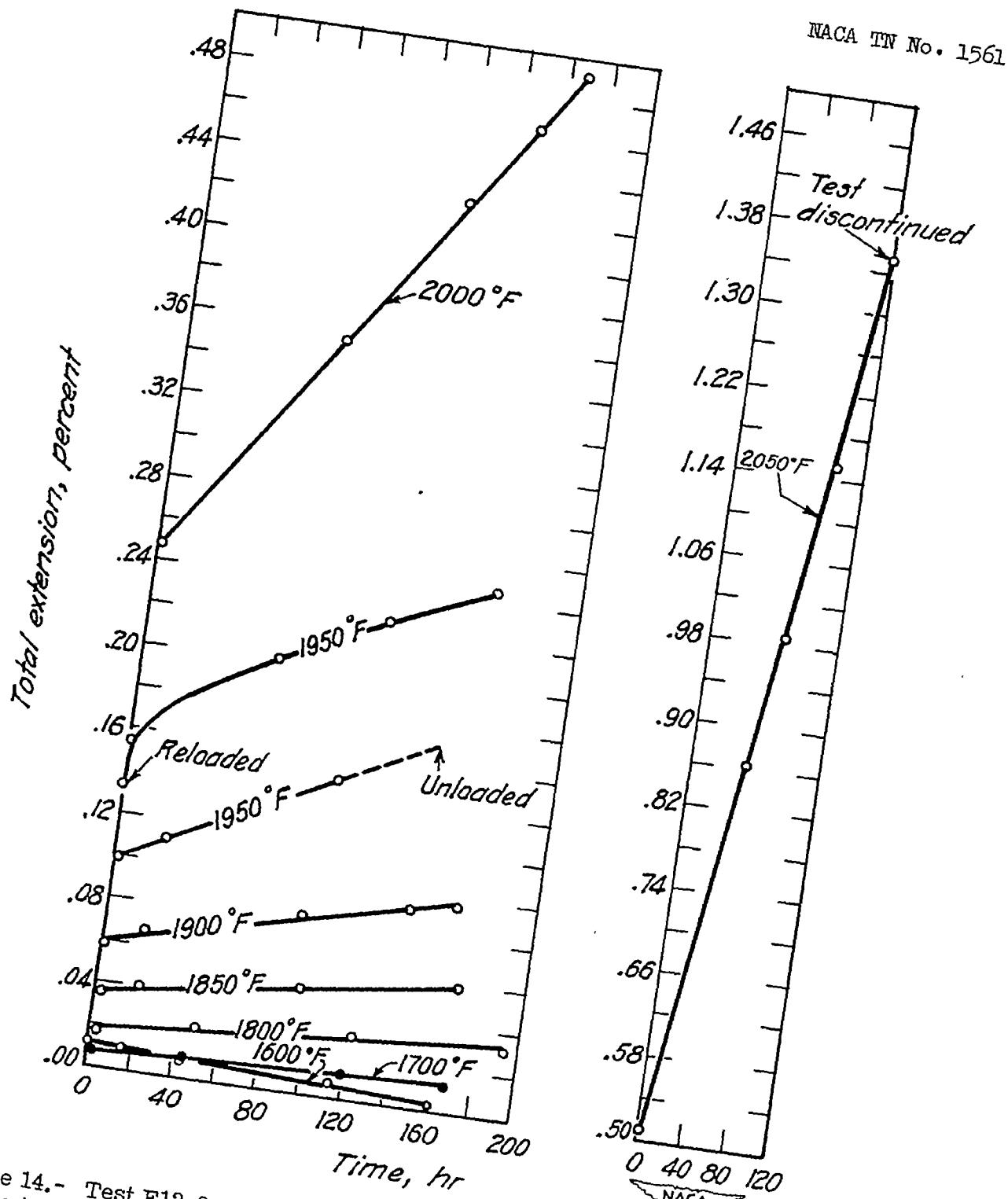
Figure 14.- Test F12-9. Time-extension data for body 151. This specimen was tested at a constant stress of 4000 psi at temperatures between 1600° and 2050° F. The test was discontinued before failure at the end of 120 hours at 2050° F.
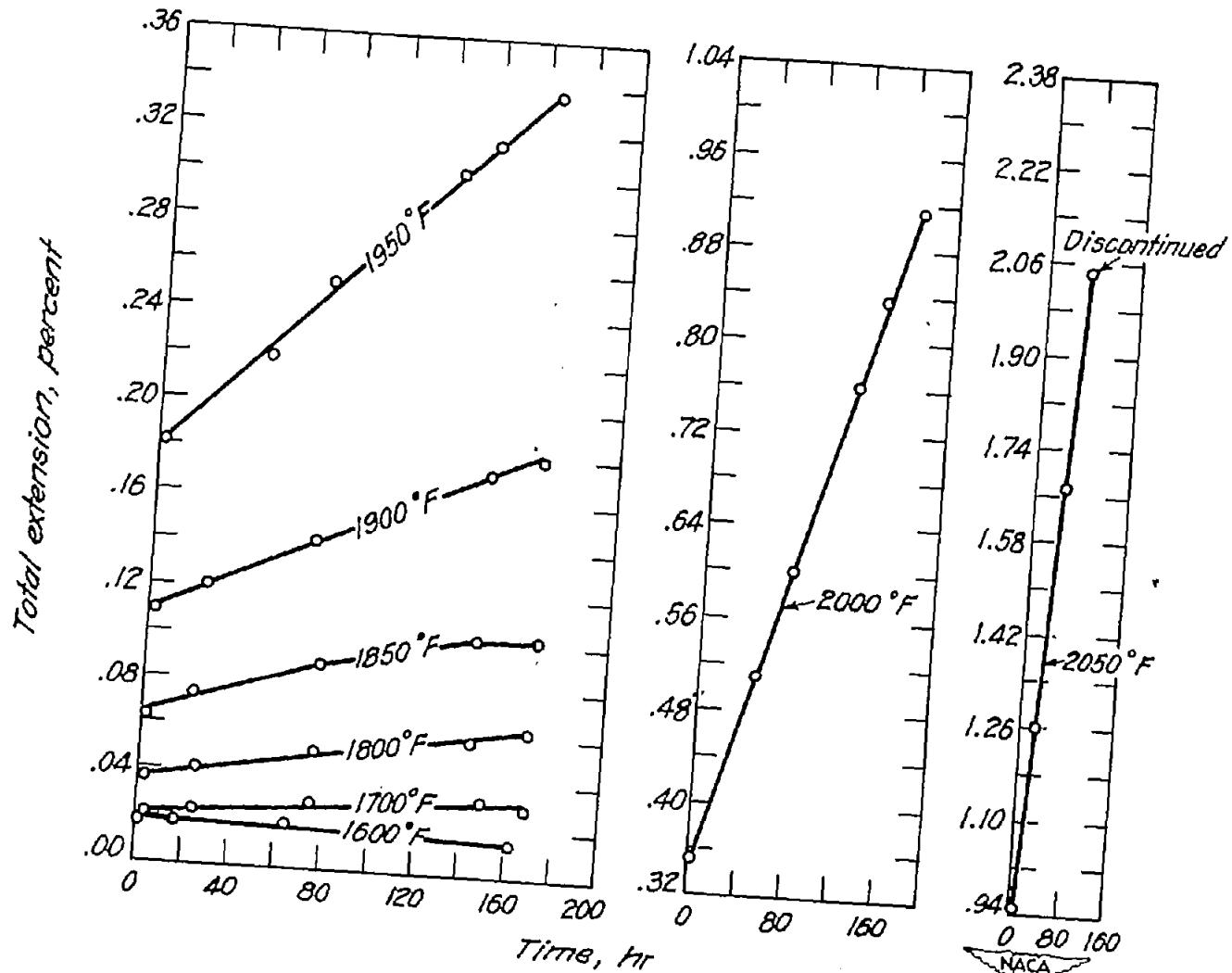
Figure 15.- Test F9-12. Time-extension data for body 151. This specimen was tested at a constant stress of 6000 psi at temperatures between 1600° and 2050° F. The temperature was not increased above 2050° F in view of the high creep rate at that temperature and the total extension of over 2 percent.
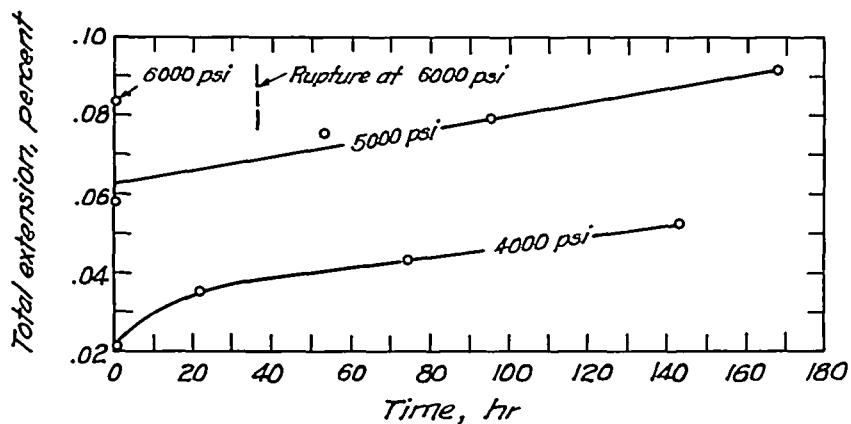
(a) Test F2-9. This specimen was tested at 1800° F at stresses between 4000 and 6000 psi. Rupture occurred within the gage length.
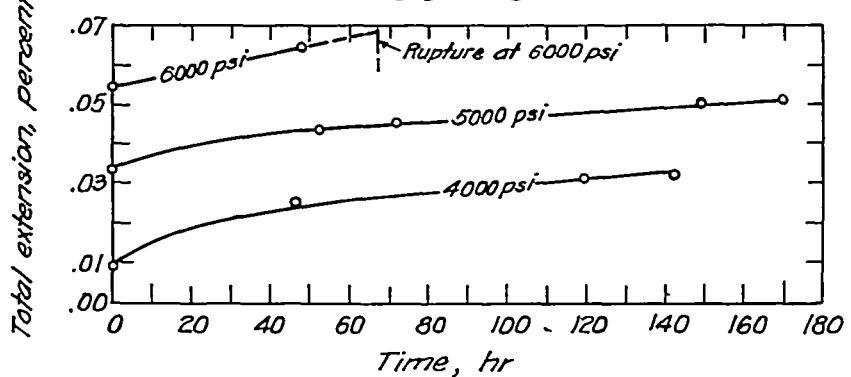
(b) Test F1-10. This specimen supported various loads at 1800° F until failure. Rupture occurred within the gage length.
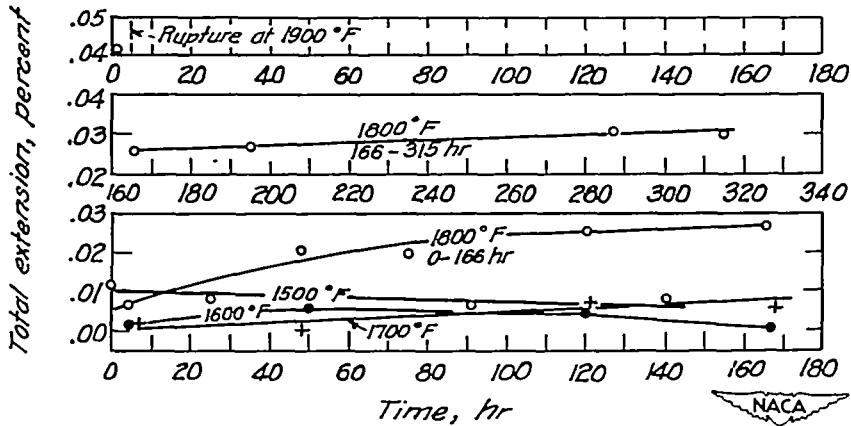
(c) Test F12-3. This specimen was tested at a constant stress of 4000 psi at temperatures between 1500° and 1900° F. Rupture occurred in the gage length.

Figure 16.- Time-extension data for body 16021T.
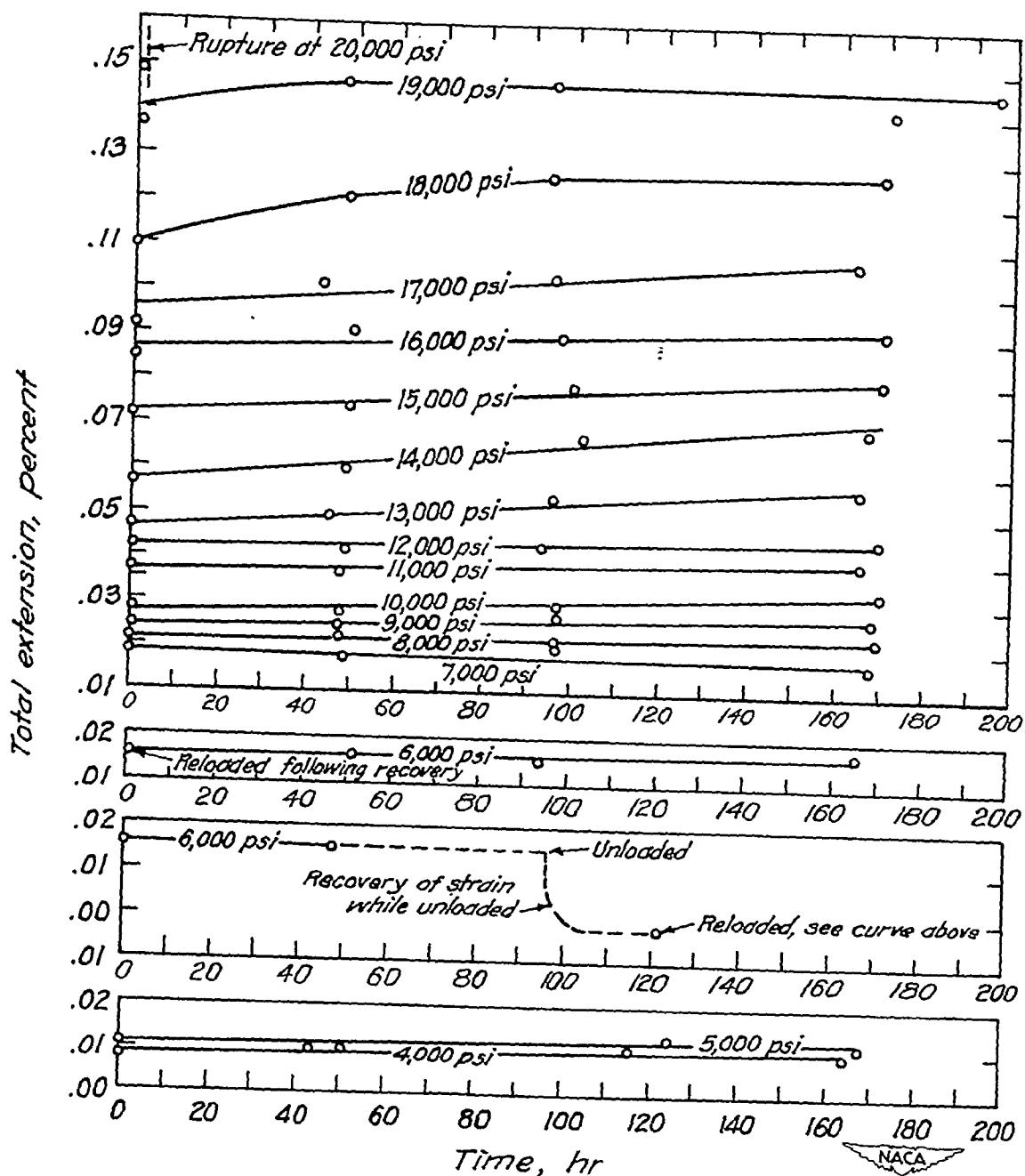
Figure 17.- Test F7-7. Time-extension data for body 4811C. This specimen was tested at 1600° F at various stresses until failure. During the testing at 6000 psi, the specimen was unloaded for 26 hours, when the power was shut off, so that the specimen would not be cooled and reheated under load. Rupture occurred near the top fillet of the specimen.
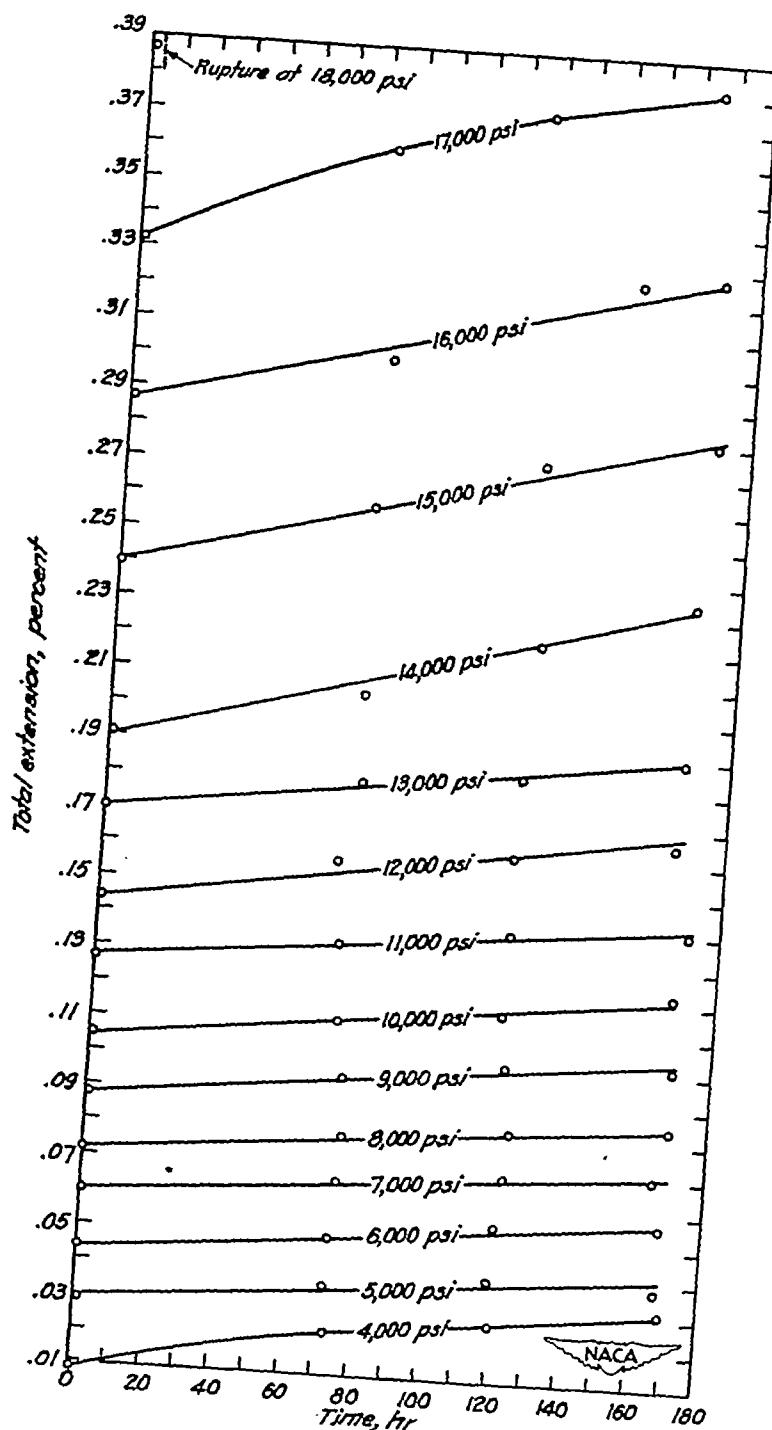
Figure 18.- Test F8-5. Time-extension data for body 4811C. This specimen was tested at 1800° F at various stresses until failure. Rupture occurred near the top fillet of the specimen, $3\frac{1}{2}$ hours after 18,000 psi was applied.
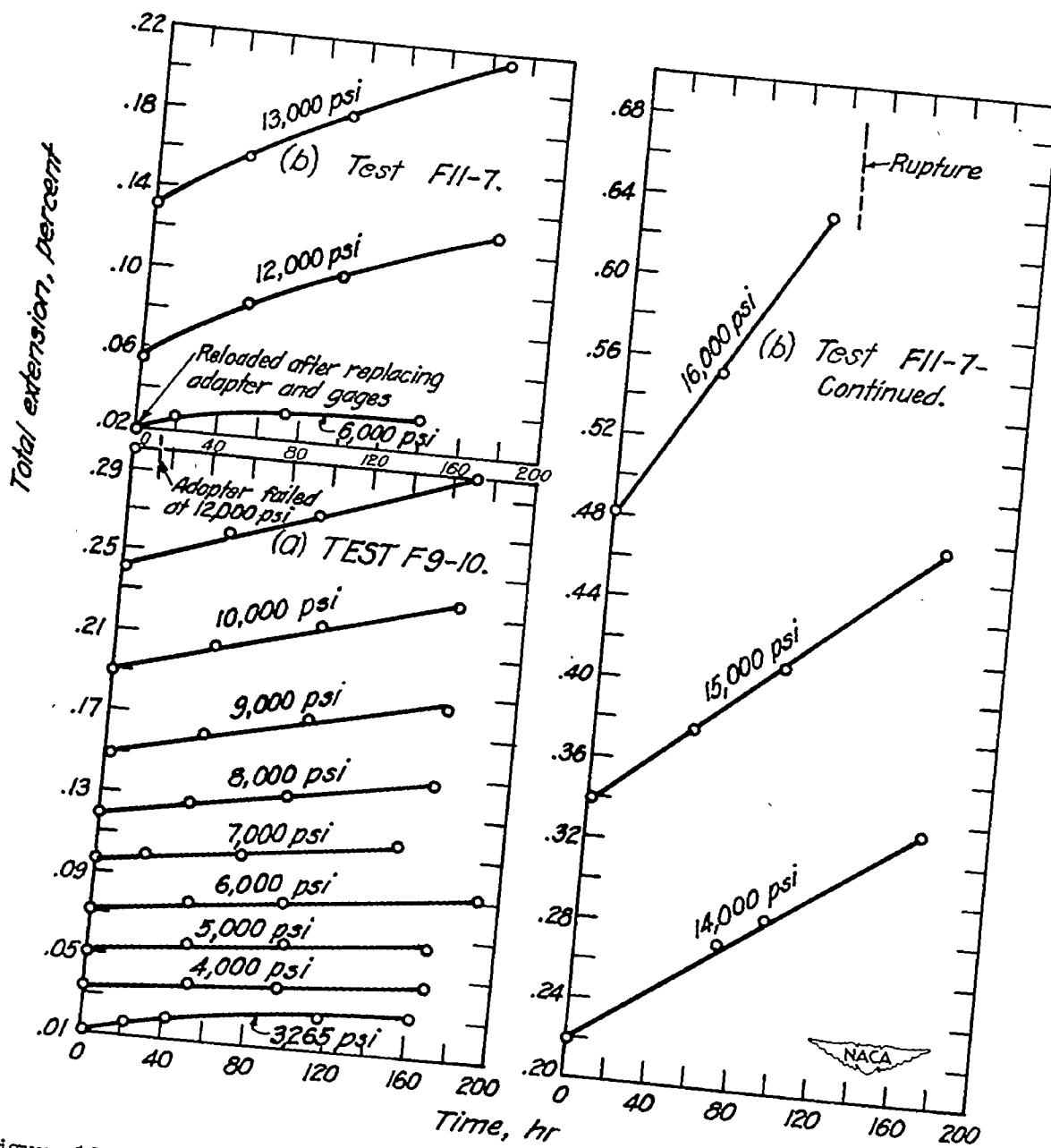
Figure 19.- Time-extension data for body 4811C. In test F9-10, the specimen was tested at a constant temperature of 1900° F at various stresses until failure. Failure occurred in the adapter, the specimen remaining intact. In test F11-7, a new adapter was fitted to the specimen used in test F9-10. The testing was continued at 1900° F at various stresses until failure. Rupture occurred within the gage length; the extensions observed during this test started from a new zero, and thus might be added to those observed in test F9-10.
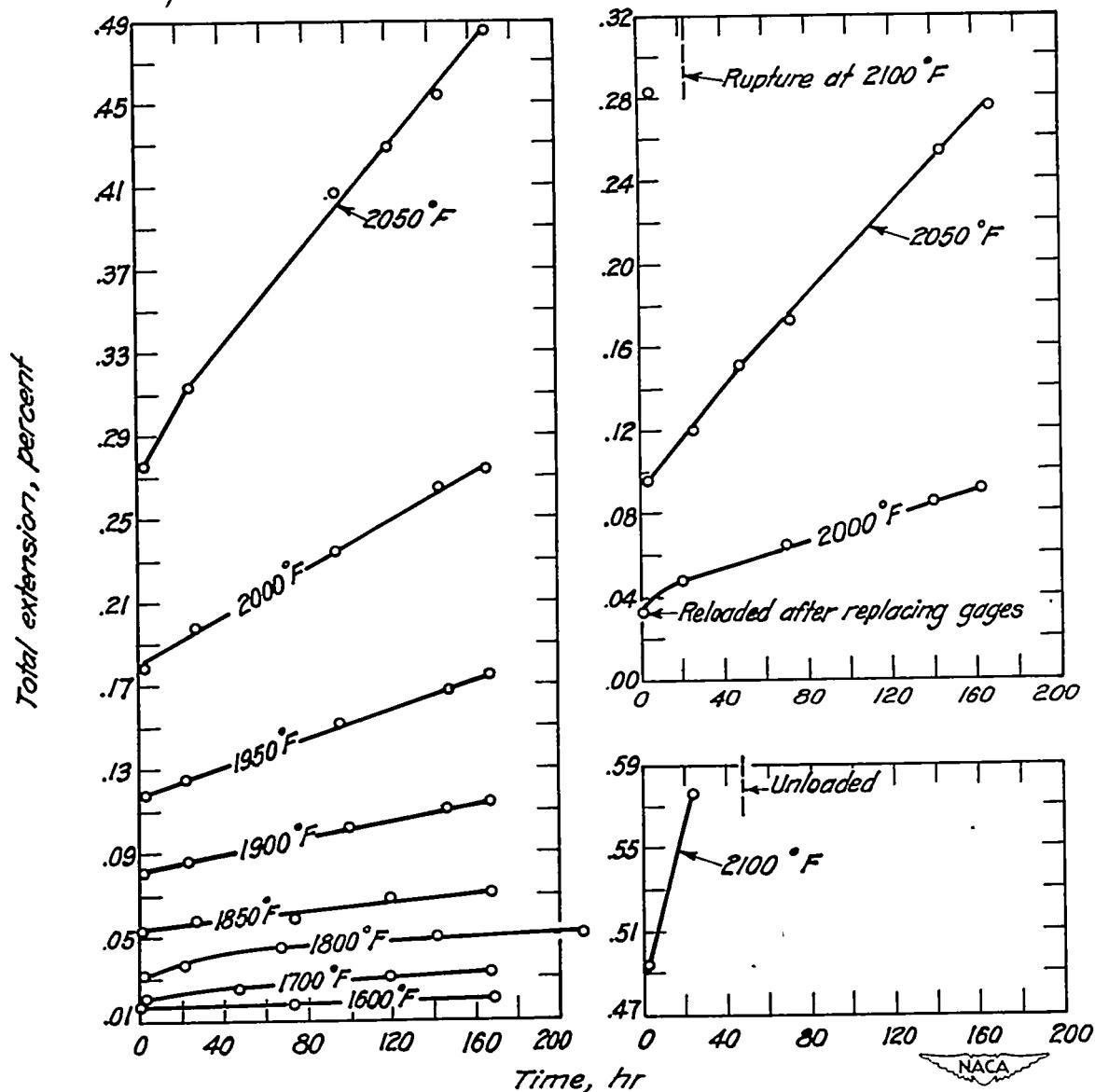
Figure 20.- Test F10-7. Time-extension data for body 4811C. This specimen
was tested at a constant stress of 6000 psi at temperatures between 1600°
and 2100° F. Failure occurred in the gage length.
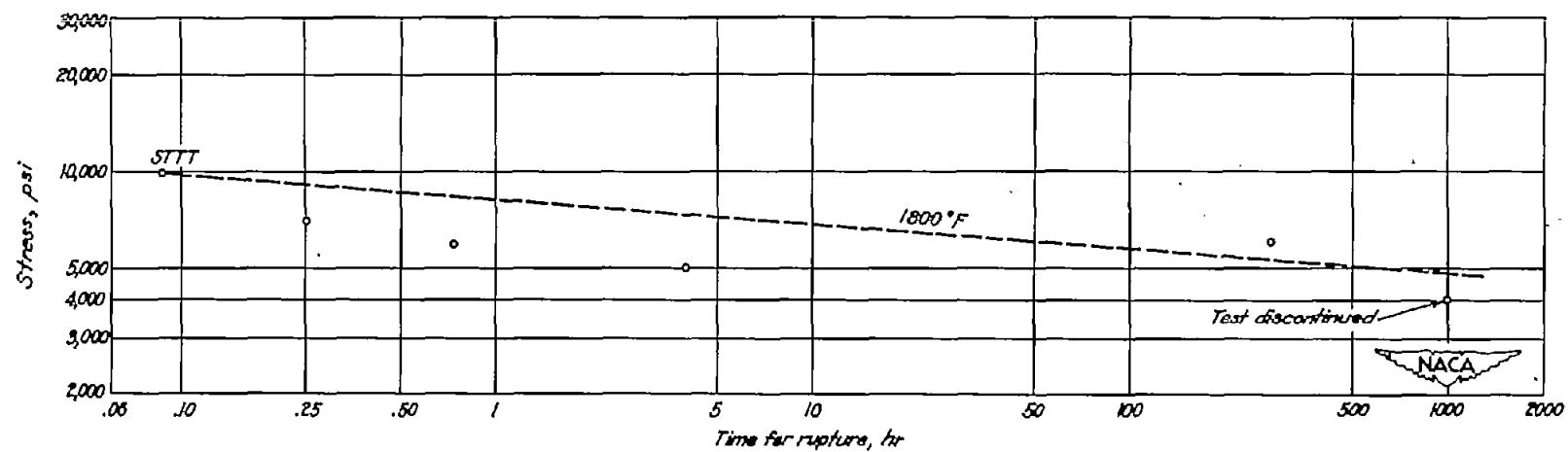
Figure 21.- Curve of stress against rupture time for body 16021T. The short-time tensile test value (STTT) was used to locate the graph, in preference to a more nearly average curve, because the higher value is believed to represent more nearly the true potentialities of the body.